



# Spectrophotometric Determination of Chromium Using Promethazine Hydrochloride - Application in Various Samples

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## Abstract

A rapid, simple and sensitive spectrophotometric method for the determination of trace amounts of chromium (VI) was studied. The method is based on the reaction of chromium (VI) with promethazine forming a red colored species by chromium in hydrochloric acid medium and exhibits a maximum absorbance at 518 nm. A plot of absorbance with chromium (VI) gives a straight line indicating that Beer's law has been obeyed over the range concentration of 0.05-4.0  $\mu\text{g/ml}$  with a molar absorptivity of chromium(VI)  $2.04 \times 10^4 \text{ l.mol}^{-1}.\text{cm}^{-1}$ , Sandell's sensitivity index of  $0.0025 \mu\text{g.cm}^{-2}$  while the limit of detection (LOD) was found to be  $0.0924 \mu\text{g.ml}^{-1}$  and a relative standard deviation of  $\pm(0.63-1.24)\%$ , depending 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, synthetic alloys and standard rock sample of known composition (MRG-1).

**Key words:** Chromium, Promethazine, Hydrochloride, Spectrophotometry.

## Introduction

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 [1].

Many spectrophotometric methods for the determination of chromium have been reported with some chromogenic reagents such as 4-(6-Nitro-2-Benzothiazolylazo)-Resorcinol[2], trifluoperazine Hydrochloride[3], Variamine blue[4], Xylene cyanol FF[5], 2-[(6-Methyl-2-Benzothiazolyl)azo]-4-Chlorophenol[6], Saccharin[7]. Many either U.V-Vis spectrophotometry method have been reported for the determination of chromium [2-9].

There are many sensitive techniques for chromium determination, such as X-ray absorption spectroscopy[10], flow-injection[11], sequential injection[12], coprecipitation with high-performance liquid chromatography (HPLC)[13], ion chromatography(IC)[14], IC-ICP-MS [15], solvent extraction and extraction of solid phase[16], ion-exchange separation[17], HPLC-ICP-MS [18], electrochemical[19], Flame Atomic Absorption Spectrometry (FAAS)[20], Inductively Coupled Plasma Mass Spectrometry (ICP-MS)[21], Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)[22], Electrothermal Atomic Absorption Spectrometry (ET-AAS)[23], Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES)[24] and fluorometry[25]. To measure the species of the chromium from the preconcentration sample, it requires a simple, safe, inexpensive optimum and short time method.

Promethazine [10-(dimethylamino) propyl phenothiazine] is a widely used drug in pharmaceutical formulation. It is being used as an antihistaminic and antiemetic drug, but it also has some antiserotonergic, anticholinergic and mark local anaesthetic properties [26]. Promethazine has been frequently used in pharmaceutical analysis; thus, it has been utilized as color producing reagent for determination of primary aromatic amines[27].

In the present investigation a facile sensitive and selective method has been reported for the determination of chromium with a new reagent promethazine hydrochloride based on the formation of a red radical cation on the oxidation of promethazine with chromium (VI) at room temperature in an acidic medium. The method has been successfully employed for the determination of chromium in various water samples, pharmaceutical preparations, synthetic alloys and a standard rock sample of known composition (MRG-1).

## Experimental

### 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 $\mu$ g/ml):** The solution is prepared by dissolving 0.2829 g of  $K_2Cr_2O_7$  and diluting to the mark with distilled water in 100 ml volumetric flask.(Fluka).

**Working Chromium (VI) solution (100 $\mu$ g/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 (10 µg/ml):** This solution was prepared by diluting 10 ml of the Working solution to 100 ml with distilled water in a volumetric flask.

**Stock .Chromium(III) solution (1000µg/ml):** The solution is prepared by dissolving 0.2829 g of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in 50 ml of water, add 1 ml saturated sodium sulfite solution, acidified with 1 ml 2.5 M sulfuric acid, and then boil for 2 minutes to remove excess SO<sub>2</sub> and dilute with water to 100 ml. Suitable volume of this solution was diluted to obtain the working standard.[28]

**Promethazine hydrochloride solution (0.2%):** The solution is prepared by dissolving 0.2 g of Promethazine (SDI) in 100 ml distilled water volumetric flask. This solution is kept in dark flask and it is stable for 7 day if it is kept in a refrigerator.

### Hydrochloric acid solution (6M)

## Result and Discussion

Under the reaction conditions, chromium was treated with promethazine reagent in acidic medium to produce a red radical cation species which exhibits absorption maxima at 518 nm against reagent blank solution .The intensity of the formed colored product has been found to be proportional to the amount of chromium originally present in the solution.

### Study of the experimental conditions:

The spectrophotometric properties of the colored product as well as the different experimental parameters affecting the color development and its stability has been carefully studied and optimized. For the subsequent experiments 10 µg of chromium are taken and final volumes are 10 ml.

### Effect of acid

To produce the colored species upon the reaction of with promethazine reagent only acid medium can achieve forming the radical cation with useful analytical properties .Therefore , different concentrations 1- 12 M hydrochloric acid solution have been tested. An intense colored product is obtained rapidly when the volume is adjusted to the mark with 6M HCl . Various acids (6M), sulfuric acid, acetic acid and formic acid have also been tried for the purpose of producing intense colored species. It was found that in the presence of these acids , maximum intensity of the color cannot be achieved , as 6M HCl , indicating the unstable nature of the colored species in these media.

### The effect of promethazine hydrochloride amount

The effect of the amount of promethazine. HCl reagent on the absorbance of the resulting colored species had next been investigated .The experimental results illustrated in (Table 1) indicated that 1 ml of 0.2 % reagent solution in the optimum volume because : (i) it shows the absorbance of the resulting product becomes independent to the chromium(VI) concentration when higher amounts of the reagent are used (ii) The blank value is low , (iii) The coefficient of determination [R<sup>2</sup> (5- 30) µg Cr (VI) ] = 0.99493 and the sensitivity of the colored reaction is high.

### Effect of Surfactants

The effect of the presence 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) surfactants on the intensity of the colored product have been studied. The experimental data Table (2) 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.

### Development Time and Stability Period

To test the effect of time on the absorbance of the colored product at the wavelength of maximum absorption at 518 nm. The colored product has been prepared from by using different amounts of chromium(VI) (5,10,20)  $\mu\text{g}$  under the optimal experimental conditions and the absorbance are measured at different intervals of time up to 60 min. The experimental results have shown that the colored product develops immediately and the absorbance remains maximum and constant for at least 50 min. The stability period for at least 50 min is sufficient for many measurements to be made.

### Effect of Masking Agents

In order to enhance the selectivity of promethazine. HCl reagent, the following general masking agents are examined ; Ethylenediaminetetraacetic acid (EDTA), Ethylene glycol-o,o'-bis(aminoethylether)-N,N,N',N'-tetra-acetic acid (EGTA), Nitritotriacetic acid (NTA), 1,2-Diaminocyclohexanetetraacetic acid (CDTA), Sodium fluoride (NaF), 5-Sulphosalicylic acid (5-SSA), Citric acid and Tartaric acid.

The results indicate that EGTA, CDTA, 5-SSA, Citric acid and Tartaric acid mask chromium, while other complexons have no effect. As a result, a composite mixture containing 1% of NTA, EDTA and NaF has been prepared. The effect of different volumes 0.5 - 3 ml of the composite mixture is tested [29].

The results indicate that 1ml of composite complexing solution is desired because of the highest intensity.

### Absorption Spectra

When a dilute solution chromium, under the above established conditions is treated with promethazine. HCl solution in the presence of 6M HCl solution a red colored product immediately formed. This shows maximum absorption at 518 nm in contrast to the reagent blank which shows maximum absorption at 355nm. Figure (1).

### Recommended Procedure and Calibration Curve

Sample aliquots containing 0.5- 40  $\mu\text{g}$  of chromium (VI) solution was placed into 10 ml volumetric flasks. To each chromium solution, 1 ml of promethazine (0.2%) was then added and volumes are made to the mark with HCl (6M). The reaction mixtures were mixed and the absorbance was measured at 518 nm, against the reagent blank prepared in the same manner but without Cr (VI). A plot of absorbance versus determinant concentration Figure (2) shows that Beer's law is obeyed over the range of (0.05- 4) ppm. The molar absorptivity being  $2.04 \times 10^4 \text{ l.mol}^{-1}\text{.cm}^{-1}$  and the Sandell's sensitivity is  $0.0025 \mu\text{g.cm}^{-2}$  while limit of detection (LOD), limit of quantitation (LOQ)[30] are found to be  $0.0924 \mu\text{g.ml}^{-1}$  and  $0.0308 \mu\text{g.ml}^{-1}$ , respectively. At higher Cr (VI) concentrations, a negative deviation occurs.

### Determination of chromium(III)

Sample aliquots containing 0.5- 40  $\mu\text{g}$  of chromium (III) solution was placed into 10 ml volumetric flasks. A volume of 0.5 ml saturated bromine water and 0.5 ml of 4M 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 then the above procedure for chromium (VI) was followed.

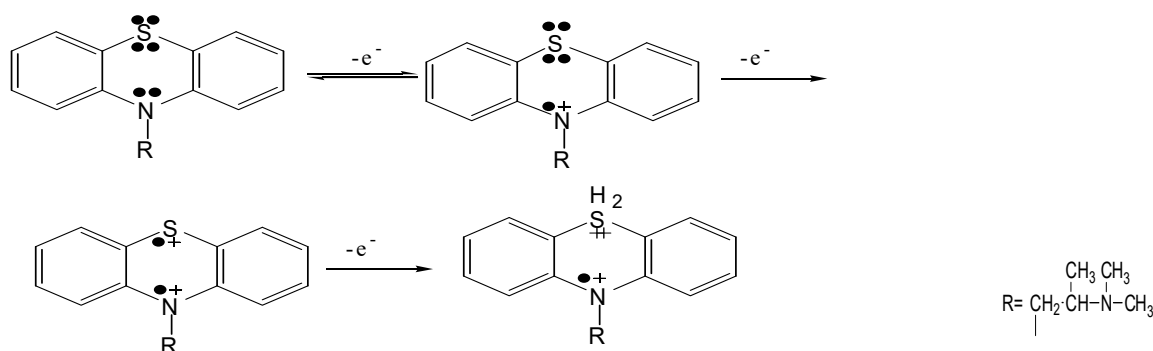
### Accuracy and Precision of the Method

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.

### Nature of reaction products

The stoichiometry of the reaction between chromium (VI) and promethazine was studied by both Job's method of continuous variations and molar-ratio method [31]. The result obtained . Figure (3) shows the existence of 1:3 (Cr : promethazine. HCl).

\* Promethazine like other phenothiazine is oxidized in an acidic medium by oxidizing agents. The oxidative reaction process is carried out in three steps [32].



### 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 10 $\mu$ g 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 Fe<sup>+2</sup>, Ce<sup>+4</sup> and V<sup>+5</sup> ions.

The effect of interfering ions Ce<sup>+4</sup>, Fe<sup>+2</sup>, V<sup>+5</sup> is studied in the presence of composite mixture containing 1% (NTA, EDTA, NaF). The results obtained are summarized in Table (5).

The effect of foreign compounds that may be present in dosage form was also studied by adding different amounts of foreign substances to 10 $\mu$ g Cr(VI) a final volume of 10 ml (1 $\mu$ g/ml). It was observed that studied foreign species did not interfere in the present method Table (6).

### 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 (North of Mosul city), Mediterranean Sea water in Turkey (Mediterranean coast, Mersin Beach Kızkalesi) and Well water (Al-qusiat near Mosul city) by the present method and the average recoveries of (10,15,20) $\mu$ g Cr(VI)/(1,2,3) ml of waters are 99.7%, 100.1%, 99.8% and 100.4% respectively.

#### II. Determination of chromium in geological reference sample (MRG -1):[33]

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 in to 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[34].The recovery is found to be 100.4% , while by the standard method the recovery is 98.1% which indicates that this method is reliable for application.

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

Samples of the finely ground multivitamin–multimineral tablets 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 H<sub>2</sub>SO<sub>4</sub>. 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).The results indicate that a good recovery was obtained for Diabetone.

### V. Determination of chromium in synthetic alloy:

Recently, the addition of Cr metal to the alloy of (Cr-Ni-Mo, Cr-Ti, Cr-Fe) has reinforced the alloy against corrosion [35].Table (8) shows the results of chromium determination in synthetic alloys.

A relatively good accuracy is shown for determining chromium in synthetic alloys.

The above results reveal that the method is suitable for determining chromium in the above samples with satisfactory recovery. Both the present method and the literature method [34] have been applied at the same time to t-test and F-test[36] and the value compared with the statistical tables for eight degrees of freedom at 95% validation level. The results in Table (9) show that there is no real difference between the two methods.

### Comparison of Methods

Table (10) shows the comparison between some of the analytical variables for the present method with that of other spectrophotometric methods.

### Conclusion

A simple, sensitive, rapid ,accurate and precise spectrophotometric determination of chromium (VI) has been carried out. The method is based on oxidation- reduction reaction between chromium(VI) and Promethazine reagent in acidic medium 6M HCl to produce a red colored product which is stable, water soluble and has maximum absorption at 518 nm. Beer's law is obeyed in the concentration range 0.05- 4 ppm with a molar absorptivity of  $2.04 \times 10^4$  l.mol<sup>-1</sup>.cm<sup>-1</sup> , Sandell's sensitivity index of 0.0025 µg.cm<sup>-2</sup> and a relative standard deviation of  $\pm 0.63$  to  $\pm 1.24$  depending on the concentration level. The present method has been developed for the determination of chromium (III).The proposed method has been applied successfully to the determination of chromium in waters, Pharmaceutical preparations, synthetic alloys and standard rock sample (MRG-1).

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**Table (1): Effect of promethazine. HCl on absorbance of colored species.**

ml of 0.2% promethazine. HCl Solution	Absorbance / $\mu\text{g}$ of Cr (VI) in 10 ml					Coefficient of determination ( $R^2$ )	Blank
	5	10	15	20	30		
0.5	0.232	0.429	0.629	0.764	0.890	0.96613	0.012
1.0	0.240	0.464	0.641	0.846	1.120	0.99493	0.018
1.5	0.236	0.456	0.681	0.896	1.188	0.99438	0.026
2.0	0.238	0.460	0.701	0.909	1.201	0.99331	0.033
3.0	0.242	0.470	0.699	0.905	1.146	0.98801	0.074

**Table (2): Effect of surfactants on absorbance and order of additions**

Surfactant Solution (2ml)	Absorbance / ml of surfactant			$\lambda_{\text{max}}$ (nm)
	I	II	III	
CPC ( $1 \times 10^{-3}$ M)	0.453	0.460	0.394	514
CTAB ( $1 \times 10^{-3}$ M)	0.446	0.452	0.410	516
SDS ( $1 \times 10^{-3}$ M)	Turbid	Turbid	Turbid	---
Triton X-100 (1% v/v)	0.436	0.451	0.435	514

\* Absorbance without surfactant = 0.469 (518 nm)

\*\* I. Cr(VI)(M) + Surfactant (S) + Reagent (R) + HCl (H)

II. M + R + S + H

III. M + R + H + S

**Table (3): Accuracy and precision of the method.**

Amount of chromium taken, ( $\mu\text{g}$ )	Recovery* (%)	RSD* (%)
5	100.5	$\pm 0.63$
10	99.8	$\pm 0.87$
20	98.5	$\pm 1.24$

\* Average of five determinations

**Table (4): Effect of interferences on the absorbance without masking agents**

Foreign ion	Form added	Error % / $\mu\text{g}$ of interference added				
		10	50	100	250	500
Al <sup>3+</sup>	AlCl <sub>3</sub> .6H <sub>2</sub> O	-0.22	-0.81	-1.32	-1.96	-2.87
Ag <sup>+</sup>	AgNO <sub>3</sub>	0.0	+0.44	-0.92	-1.78	-2.02
Ba <sup>2+</sup>	BaCl <sub>2</sub> .2H <sub>2</sub> O	+0.26	0.0	+0.86	+1.55	+3.06
Be <sup>2+</sup>	BeSO <sub>4</sub> .4H <sub>2</sub> O	0.0	+0.67	-1.27	-1.87	-2.26
Bi <sup>3+</sup>	Bi(NO <sub>3</sub> ) <sub>3</sub> .5H <sub>2</sub> O	-0.85	+1.04	-1.50	-2.14	-3.49
Ca <sup>2+</sup>	CaCl <sub>2</sub>	0.0	+0.47	-0.83	-1.32	-2.30
Cd <sup>2+</sup>	Cd(CH <sub>3</sub> COO) <sub>2</sub> .H <sub>2</sub> O	+0.43	0.0	+0.72	-1.09	-1.89
Ce <sup>4+</sup>	Ce(NH <sub>4</sub> ) <sub>4</sub> (SO <sub>4</sub> ) <sub>4</sub> .2H <sub>2</sub> O	+31.0	+56.12	+73.96	+89.88	+106.27
Co <sup>2+</sup>	Co(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	+0.21	0.0	+0.52	+0.99	+1.98
Cs <sup>+</sup>	CsNO <sub>3</sub>	0.0	-0.22	-0.82	+1.65	-2.21
Cu <sup>2+</sup>	CuSO <sub>4</sub>	-0.20	+0.56	+1.31	-1.96	-2.74
Fe <sup>2+</sup>	FeCl <sub>2</sub>	-20.17	-41.74	-70.83	-81.69	-97.61
Fe <sup>3+</sup>	Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	-0.83	-1.21	-1.98	+2.36	+3.13
Hg <sup>2+</sup>	Hg(NO <sub>3</sub> ) <sub>2</sub>	+0.81	-1.10	-1.45	+2.10	+3.28
K <sup>+</sup>	KCl	0.0	-0.84	-1.89	+1.31	-2.18
La <sup>3+</sup>	La(NO <sub>3</sub> ) <sub>3</sub> .6H <sub>2</sub> O	-0.44	-0.87	-1.09	-1.91	-2.71
Li <sup>+</sup>	Li <sub>2</sub> SO <sub>4</sub> .H <sub>2</sub> O	-0.62	-0.41	+0.88	-1.26	-2.43
Mg <sup>2+</sup>	MgCl <sub>2</sub> .6H <sub>2</sub> O	0.0	+0.64	+1.01	+1.32	+1.82
Mn <sup>2+</sup>	Mn(NO <sub>3</sub> ) <sub>2</sub> .4H <sub>2</sub> O	0.0	-0.41	-0.95	-1.45	-2.30
Mo <sup>6+</sup>	(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> .4H <sub>2</sub> O	-0.45	-0.96	+1.51	-2.12	-3.38
Na <sup>+</sup>	NaCl	+0.66	0.0	-1.54	-1.76	-2.21
Ni <sup>2+</sup>	Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	0.0	+0.41	+0.66	+1.10	-2.04
Pb <sup>2+</sup>	Pb(NO <sub>3</sub> ) <sub>2</sub>	+0.21	-0.66	-1.10	-1.31	-2.65
Pd <sup>2+</sup>	PdCl <sub>2</sub>	+0.45	-0.85	+1.87	-2.08	+3.34
Sc <sup>3+</sup>	Sc <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .6H <sub>2</sub> O	+0.46	+0.88	+1.52	-1.90	-3.29
Se <sup>2+</sup>	SeO <sub>2</sub>	-0.34	-0.55	-0.85	+1.33	+2.10
Sn <sup>2+</sup>	SnCl <sub>2</sub> .2H <sub>2</sub> O	-0.62	-0.98	-1.75	-2.02	-2.96
Sr <sup>2+</sup>	SrCl <sub>2</sub> .6H <sub>2</sub> O	-0.43	+0.66	-0.88	+1.53	-2.17
Te <sup>4+</sup>	TeO <sub>2</sub>	+0.45	-0.71	+1.66	+1.42	-3.18
Th <sup>3+</sup>	Th(NO <sub>3</sub> ) <sub>4</sub> .6H <sub>2</sub> O	+0.78	+0.94	+1.20	-2.22	+2.84
Ti <sup>3+</sup>	TiCl <sub>3</sub>	+0.22	-0.88	-1.61	-2.04	+2.38
V <sup>5+</sup>	NH <sub>4</sub> VO <sub>3</sub>	+22.15	+52.78	+76.81	+99.82	+128.71
W <sup>6+</sup>	Na <sub>2</sub> WO <sub>4</sub> .2H <sub>2</sub> O	+0.87	-1.32	-1.90	-1.66	-3.60
Y <sup>3+</sup>	YCl <sub>3</sub> .6H <sub>2</sub> O	+0.21	+1.10	+1.25	+1.81	+2.20
Zn <sup>2+</sup>	Zn(Ac) <sub>2</sub> .2H <sub>2</sub> O	0.0	+0.62	-0.97	-1.54	-2.92
Zr <sup>4+</sup>	ZrOCl <sub>2</sub> .8H <sub>2</sub> O	-0.45	-1.02	-1.88	-2.29	-3.66
F <sup>-</sup>	NaF	0.0	+0.76	+1.38	+1.90	+2.06
Cl <sup>-</sup>	NaCl	+0.22	-0.89	-1.32	-1.56	-2.89
Br <sup>-</sup>	KBr	+0.45	+0.99	+1.76	+2.04	+3.23
SO <sub>3</sub> <sup>2-</sup>	Na <sub>2</sub> SO <sub>3</sub>	-0.95	-1.26	-1.60	-1.96	-2.53
SO <sub>4</sub> <sup>2-</sup>	Na <sub>2</sub> SO <sub>4</sub>	+0.62	+0.98	+1.51	+1.98	+2.04
CO <sub>3</sub> <sup>2-</sup>	Na <sub>2</sub> CO <sub>3</sub>	0.0	+0.43	+0.91	+1.26	+1.95
HCO <sub>3</sub> <sup>-</sup>	NaHCO <sub>3</sub>	-0.38	+0.52	-0.76	-1.09	+1.59
NO <sub>3</sub> <sup>-</sup>	NaNO <sub>3</sub>	-0.61	+0.74	-1.64	-1.91	-3.13
PO <sub>4</sub> <sup>3-</sup>	NaH <sub>2</sub> PO <sub>4</sub> .2H <sub>2</sub> O	-0.43	-1.09	-1.43	-1.65	-2.22
BO <sub>3</sub> <sup>3-</sup>	H <sub>3</sub> BO <sub>3</sub>	-0.44	-0.71	+1.42	-1.96	-3.41
ClO <sub>4</sub> <sup>-</sup>	HClO <sub>4</sub>	+0.20	+0.83	+1.14	-1.46	-2.45

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

Foreign ion	Form added	Error % $\mu\text{g}$ of interference added				
		5	10	25	50	< 100
$\text{Ce}^{4+}$	$\text{Ce}(\text{NH}_4)_4(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$	+2.0	+4.02	+8.18	+21.84	+44.28
$\text{Fe}^{2+}$	$\text{FeCl}_2$	-1.12	-1.88	-2.72	-5.02	-19.98
$\text{V}^{5+}$	$\text{NH}_4\text{VO}_3$	+11.60	+34.52	+45.11	+85.17	+99.25

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

Interferences	Recovery(%) of $10\mu\text{g}$ Cr (VI)/ ppm of interfere added		
	100	250	500
Acacia	100.3	98.6	101.3
Glucose	99.5	100.6	98.3
Lactose	99.7	101.2	100.8
Starch	100.0	99.3	98.5

**Table (7): Analytical results of Cr in Pharmaceutical Samples.**

Drug	Sample ( $\mu\text{g}/\text{ml}$ )	Recovery*(%)	Recovery*(%) Standard method
Diabetone (Vitabiotics.com.), London, England	5	99.2	99.6
	10	99.5	99.2
	20	98.5	98.5
Century (Multivitamin & Multimineral),USA	5	86.9	98.3
	10	79.4	96.5
	20	71.2	95.7

**Table (8): Determination of chromium in synthetic alloys**

Alloy	Recovery, (%)	Recovery*(%) Standard method
$10\mu\text{g}$ Cr, $10\mu\text{g}$ Ni, $10\mu\text{g}$ Mo	100.6	100.2
$10\mu\text{g}$ Cr, $100\mu\text{g}$ Ni, $100\mu\text{g}$ Mo	98.5	98.5
$10\mu\text{g}$ Cr, $10\mu\text{g}$ Ti	99.3	99.2
$10\mu\text{g}$ Cr, $100\mu\text{g}$ Ti	101.0	98.1
$10\mu\text{g}$ Cr, $10\mu\text{g}$ Fe	99.5	99.8
$10\mu\text{g}$ Cr, $100\mu\text{g}$ Fe	101.4	102.2

**Table (9): The result of t-test and F-test.**

Samples	Cr(VI) ( $\mu\text{g}$ )	Recovery %*		t.exp	F.exp
		Present method	Literature method [41]		
MRG -1	10	99.0	98.5	0.98	2.00
Drug (Diabetone)	10	99.5	99.2	0.28	1.92
Cr, Ni ( $10\mu\text{g}$ ), Mo ( $10\mu\text{g}$ )	10	100.6	100.2	0.36	2.42
Cr, Ti ( $10\mu\text{g}$ )		99.3	99.2	0.78	1.60
Cr, Fe ( $10\mu\text{g}$ )		99.5	99.8	0.59	1.14

\* Average of three determinations.

**Table (10): Comparison of the methods**

Analytical parameter	Present method	Literature method <sup>[3]</sup>	Literature method <sup>[37]</sup>
Reagent	Promethazine. HCl	Trifluoperazine. HCl	bis(salicylaldehyde) orthophenylenediamine (BSOPD).
The reaction medium	Acidic	Acidic	Acidic
$\lambda_{\max}$ (nm)	518	505	478
Stability period	At least 50 min	2hour	6 hour
Beer's law range (ppm)	0.05- 4.0	0.2 -1.8	0.02- 4.0
Molar absorptivity ( $\text{l.mol}^{-1}.\text{cm}^{-1}$ )	$2.04 \times 10^4$	$2.08 \times 10^4$	$1.67 \times 10^4$
Sandell's sensitivity ( $\mu\text{g}.\text{cm}^{-2}$ )	$2.5 \times 10^{-3}$	$6.849 \times 10^{-3}$	$1.0 \times 10^{-3}$
detection limit (DL) $\mu\text{g}.\text{ml}^{-1}$	0.0924	0.0033	---
RSD (%)	$\pm (0.63-1.24)$	---	----
Application of the method	various water samples and Pharmaceutical preparations and synthetic alloys and standard rock sample of (MRG-1).	alloy steels, industrial effluents and pharmaceutical samples.	industrial, environmental, biological and soil samples

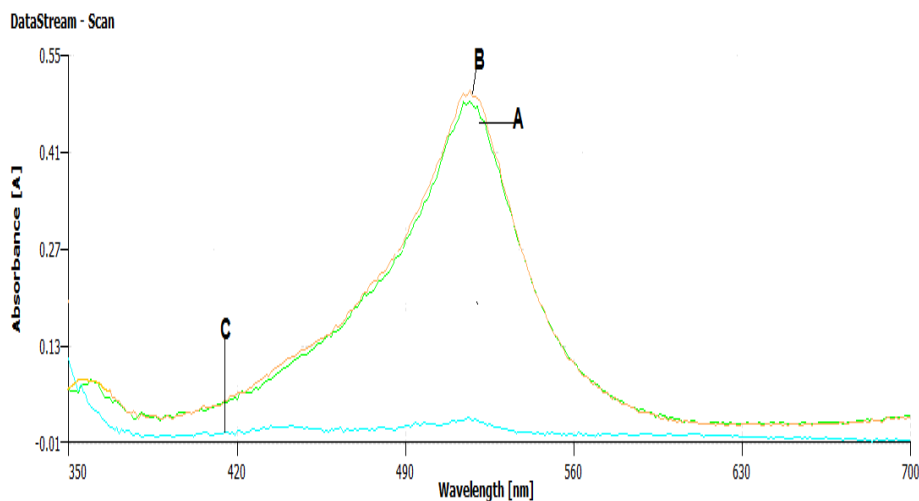
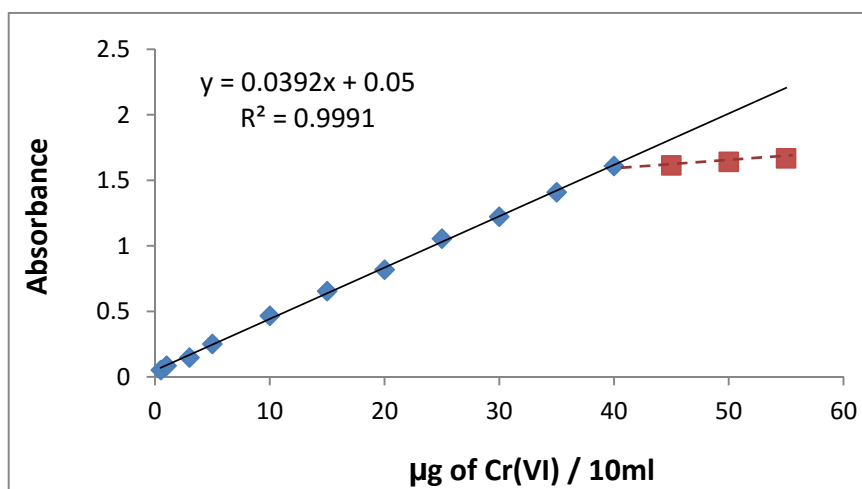
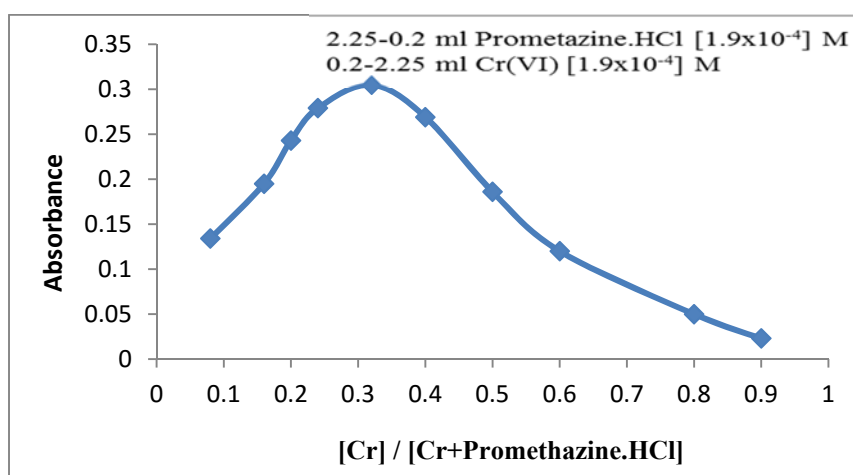


Figure (1): Absorption spectra of 10 µg Cr (VI) / 10 ml measured against (A) blank, (B) distilled water and (C) blank against distilled water.



Figure(2): Calibration curve for chromium determination with promethazine reagent.



Figure(3): Continuous variation slop for chromium (VI) with promethazine. HCl reagent.



## التقدير الطيفي للكروم باستعمال كاشف البروميثازين هيدروكلوريد - 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 كاشف البروميثازين هيدروكلوريد الذي يكون جذراً حراً موجباً ذا لون احمر عند اكسدته بالكروم السداسي في الوسط الحامضي لحمض الهيدروكلوريك الذي اعطى اعلى امتصاص عند طول موجي مقداره 518 نانوميتر. اظهر منحنى الامتصاص مقابل التركيز خطاً مستقيماً والذي يؤكد على انه يتبع قانون بير بمدى التراكيز من 0.05 الى 4.0 مايكروغرام. مللتر<sup>-1</sup> من الكروم السداسي وكانت الامتصاصية المولارية  $2.04 \times 10^4$  لتر.مول<sup>-1</sup>.سم<sup>-1</sup> ودلالة ساندل للحساسية 0.0025 مايكروغرام.سم<sup>-2</sup> وحد الكشف 0.0924 مايكروغرام. مللتر<sup>-1</sup> , والانحراف القياسي النسبي بين (±0.63) و (±1.24) % اعتماداً على مستوى التركيز. وقد طورت الطريقة المقترحة لتقدير الكروم الثلاثي وتم تطبيق الطريقة بنجاح لتقدير الكروم في نماذج مائية مختلفة ومستحضرات دوائية وفي نموذج من السبائك المصنعة وكذلك في نموذج من الصخور القياسية (MRG-1).

الكلمات المفتاحية: الكروم , بروميثازين. هيدروكلوريد , المطيافية .