DOI: http://dx.doi.org/10.21123/bsj.2018.15.2.0181

Spectrophotometric Determination of Sulphite in Various Water Samples Via Chromium-1,5-Diphenylcarbazide Complex

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Received 21/1/2018, Accepted 22/4/2018, Published 3/6/2018

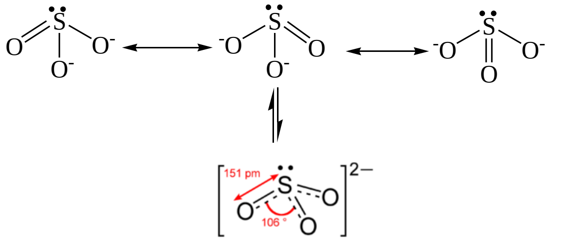
Abstract:

A simple and rapid spectrophotometric method for the determination of sulphite SO$_3^{-2}$ is described. The method is based on the rapid reduction of known amount of chromate CrO$_4^{-2}$ in the presence of sulphite in acidic medium of 2N H_2SO_4. The amount of excess of chromate was measured after it reactions with 1,5-diphenylcarbazide which finally gives a pink-violet, water soluble and stable complex, which exhibit a maximum absorption at 542 nm. Beer's law was obeyed in the concentration range from 0.004-6.0 µg of sulphite in a final volume of 25 ml with a molar absorbtivity of 4.64×10^4 l.mol^{-1}.cm^{-1}, Sandal's sensitivity index of 0.001724 µg .cm^{-2} and relative standard deviation of ±0.55 - ±0.83 depending on the concentration level. The present method has been developed for the determination of sulphite in the presence of thiosulphate. Good recoveries of sulphite from various water samples are achieved using the proposed method.

Keywords: Sulphite, Spectrophotometic method, Chromium (III), Chromate-DPC, 1,5-diphenylcarbazide.

Introduction:

Sulfites (also sulphites) are compounds that contain the sulphite ion SO$_3^{-2}$ (additive IUPAC name: trioxidosulphate (2−)). The structure of the sulphite anion can be described with three equivalent resonance structures, the structure predicted by VSEPR theory is trigonal pyramidal, as in ammonia (NH_3). In the hybrid resonance structure, the S-O bonds are equivalently of bond order one and one-third (1). Scheme (1).



Scheme 1. Equivalent resonance structure of sulphite ion.

Sulphites are some of the oldest and most widespread preservatives in our food supply. They were used in greek and roman times in wine, but it was only in the 1880s that their use in as preservatives in meats was pioneered by australian and south american beef producers wanting to ship their products to england. The use of sulphites in fruit and vegetables became common with the growth of the processed food industry in the twentieth century (2).

Sulphites destroy thiamine (Vitamin B$_1$) so some experts recommend that foods which are a significant source of thiamine, such as meats, dairy foods. Sulphites are also thought to destroy folic acid (3). High blood and urine levels of sulphite in babies can be caused by molybdenum cofactor deficiency disease which leads to neurological damage and early death (4).

Several methods have been used for the determination of sulphite, such as applying of flow injection analysis for determining sulphites in food and beverages (5). determination of sulphite in dried garlic by reversed-phase ion-pairing liquid chromatography with post-column detection has been reported (6), a capillary isotachophoretic method of sulphite (free, bound and total) determination in mustard is described (7). Also spectrophotometric methods have been used for the determination of sulphite. The 5,5′-dithiobis(2-nitrobenzoic acid) and para-rosaniline-formaldehyde methods were rapid for the determination of sulfite since it only needed one main reagent and the methods were robust and easy to operate (8), other method determination sulphite at nanogram level in alcoholic and nonalcoholic beverage samples based on diffuse reflectance fourier transform infrared spectroscopic analysis on

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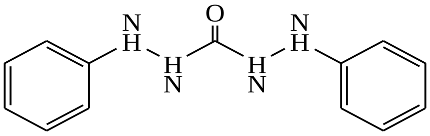
KBr matrix(9),or bleaching colour of malachite green by sulphite(10). A flow-injection is used for the determination of sulphite in white wines involving gas diffusion through a concentric tubular membrane(11). Other spectrophotometricmethod has been used for determination of sulphide, sulphite and ethanol involving gas diffusion(12), or quantification of sulphite ions in environmental samples(13).

1,5-Diphenyl carbazide (scheme 2), (DPC) is an organic compound usually used in analytical chemistry for colorimetric measurements. It exhibits many useful properties. It is used as an artificial donor during charge separation in photochemical reactions and also photosynethesis electron transport.



1,5-Diphenyl carbazide
$C_{13}H_{14}N_4O$
M.wt. = 242.28

Scheme 2. 1,5-Diphenyl carbazide structurre

It is well known that chromate-1,5-diphenylcarbazide chelate shows an intense pink-violet colour at pH 0.2 (14) on the other hand sulphite reduced chromate to chromium (III) then the excess of chromate reacted with 1,5-diphenylcarbazide. The purpose of this work is to make use of these fact to develop a simple, sensitive and rapid spectrophotometric method for the determination of sulphite, without requiring an expensive instrumentation, needless of extraction and temperature control, and the possibility of application of the proposed method to determination of sulphite in water samples.

Materials and Methods:

Apparatus

Spectral and absorbance measurements are carried out using shimadzu UV-160, UV-Visible computerized double-beam spectrophotometer. In all measurements 1-cm matched cells are used. The pH measurements are carried out using HANA pH meter.

Chemicals

All chemicals used are of analytical reagent grade.

Standard sodium sulphite solution: An approximately 0.1 N solution is prepared by dissolving a known amount of sodium sulphite anhydrous (Fluka) in distilled water containing 2 ml of 0.01% D(-)fructose as a stabilizer. This solution is standardized by iodimetry. Working standard sulphite solution with (0.01% D(-) fructose) (15), are prepared by suitable dilutions of the standard with distilled water. A 100 ppm standard sulphite solution is stable for at least 3 days.

Potassium chromate solution 4.31×10^{-4} M:

This solution was prepared by dissolving 0.0837 gm from potassium chromate (Fluka) in 100 ml distilled water in a volumetric flask. The solution was transferred to a dark bottle and it stable for at least one month.

1,5-diphenylcarbazide solution 1×10^{-3} M:

This solution was prepared by dissolving 0,0605 gm of 1,5-diphenylcarbazide (BDH) in enough amount of pure acetone, then complete the volume to the mark with 250 ml distilled water in a volumetric flask.

Sulphuric acid 2N:

This solution was prepared by appropriate dilution of 98% concentration sulphuric acid solution to the mark with 250 ml distilled water in a volumetric flask.

Masking agent solution:

This solution was prepared by dissolving 0.4778 g of nitrilotriacetic acid (NTA) in 200 ml distilled water, using 2M NaOH for effecting the dissolution. After dissolution, 0.9356 g of EDTA (disodium salt dehydrate) was added. The pH of the solution was brought to 0.2 with 0.2 M HCl and the volume was complete to the mark with 250 ml of distilled water in a volumetric flask.

Pure acetone.

Recommended procedure for the determination of sulphite:

Aliquots of standard solution 0.1-150 µg of sulphite was transferred in to a series of 25 ml volumetric flask. To each flask, 3 ml of masking agent solution 1 ml of chromate solution 4.31×10^{-4} M, 2.5 ml of sulphuric (2N) acid and 2 ml of 1,5-diphenylcarbazide reagent (DPC) 1×10^{-3} M, were added. The solution was diluted to the mark with distilled water and mixed well after 2 min, the absorbance was measured against a similarly prepared reagent blank at 542 nm using 1-cm cells. The colour was stable for at least 3.0 hr.

Results and Discussion:

For subsequent experiments, 5 µg of sulphite were taken and the final volumes are 25 ml.

Colour reaction:

In aqueous solution chromate reduced in acid medium by sulphite, then the unreduced chromate was reacted with the reagent DPC to give the an intense pink-violet complex. The reaction sequence is postulated as follows (scheme 3):

$$2\,CrO_4^{-2} + 2\,H^+ \longrightarrow Cr_2O_7^{-2} + H_2O$$

$$Cr_2O_7^{-2} + SO_3^{-2} \longrightarrow 2\,Cr^{+3} + SO_4^{-2} + \underset{\text{Unreduced}}{Cr_2O_7^{-2}}$$

$$2K_2CrO_4 + 5H_2SO_4 + 3Na_2SO_3 \longrightarrow 2K_2SO_4 + Cr_2(SO_4)_3 + 3Na_2SO_4 + 5H_2O$$

$$\underset{\text{Unreduced}}{Cr_2O_7^{-2}} + \text{1,5-Diphenyl carbazide} \longrightarrow \text{Pink-violet complex}$$

Scheme 3. The reaction sequence of spectrophotometric of sulphite

Absorption spectra

When sulphite was treated according to the recommended procedure, the absorption spectrum, show in Fig.1, was obtained. The sample solution shows maximum absorption at 542 nm, characteristic of the chromate-DPC chromophore, in contrast to the reagent blank which shows slight absorption at this wavelength, emphasising the need for measurements to be performed against the reagent blank.

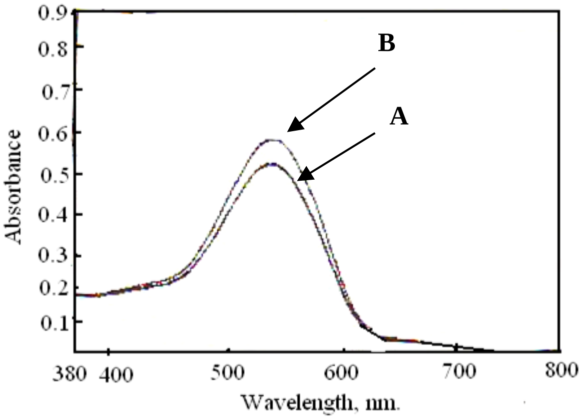


Figure 1. Absorption spectra of 5 µg of SO_3^{-2}/25 ml measured against (A) reagent blank, (B) Chromate against reagent blank.

Study of optimum conditions:

The effect of various parameters on the intensity of the coloured system have been studied and optimum conditions have been selected.

Effect of sulphuric acid volume

In order to choose the optimum amount of sulphuric acid on the reaction of sulphite ions with chromate, and the formation of a stable coloured complex between chromate and DPC, different amounts (0-5.0) ml of 2N sulphuric acid were tested. The results are shown in Table 1 indicate that 2.5 ml of 2N H_2SO_4 is considered optimum, as it gives the more stable coloured complex [11], therefore it was recommended for subsequent experiments.

Table 1. Effect of sulphuric acid.

ml of 2N sulphuric acid	Absorbance	Final pH
0.0	0.346	1.78
0.1	0.421	1.44
0.3	0.488	1.11
0.5	0.522	0.93
1.0	0.561	0.53
1.5	0.563	0.46
2.0	0.565	0.34
2.5	0.564	0.23
3.0	0.568	0.17
4.0	0.566	0.13
5.0	0.569	0.11

Effect of time

The effect of different time amount on the reducing chromate CrO_4^{-2} to Cr(III) by sulphite ions on the absorbance of the resulting coloured complex has been investigated. The results showed that 2 min reaction time was optimum and recommended for the subsequent experiment.

Effect of 1,5-diphenylcarbazide volume

It was found from the experimental results that 2 ml of DPC 1×10^{-3}M was optimum (correlation coefficient = 0.9903), and recommended for the subsequent experiment.

Effect of surfactants

The presence of surfactants in a coloured mixture solution frequently does not lead to an increase in the absorbance or a shift in the wavelength to higher values. Then a test of the effect of surfactant addition to the reaction mixture. In this respect, sodium dodecyl sulfate (SDS) (anionic surfactant), cetyltrimethyl-ammonium bromide (CTAB), cetylpyridinium chloride (CPC) (cationic surfactants) and Triton X-100 (non-ionic surfactant) have been introduced. The results indicated that addition of surfactants gave no useful effect. Therefore, omitted in this study.

Effect of masking agents

Masking agents including ethylenediaminetetraacetic acid (EDTA), nitrilotriacetic acid (NTA), trans-1,2-diamino-cyclohexane-N,N,N',N'-tetraacetic acid (CDTA) and ethyleneglycol-o,o'-bis(2-aminoethylether) -N,N,N',N'-tetraacetic acid (EGTA) were examined for increasing the selectivity of the suggested method. The results indicate that CDTA and EGTA give no useful results. Other complexions (EDTA and NTA) have no remarkable effect, therefore, composite masking solution containing 0.01 M of each one has been prepared, and added in portions from 1-5 ml. The 3 ml of composite masking solution is selected for the subsequent experiments. The purpose of used composite masking solution was to make the method seemed to be moderately selective in the presence of composite masking solution.

Effect of order of addition:

The different orders of addition were studied. The results shown in Table 2 indicate that the first order was optimum because it gives lowest absorbance value, therefore recommended for the subsequent experiment.

Table 2. Effect of order of addition

Reaction component	Order number	Absorbance
S+M.A+C+H+R	I	0.522
S+H+M.A+C +R	II	0.529
S+R+H+M.A+C	III	0.526
S+H+R+C+M.A	IV	0.533
S+C+M.A+R+H	V	0.527
S+R+C+M.A+H	VI	0.531

S= Sulphite, M.A = Masking agent, C=Chromate, H= Sulphuric acid, R= DPC.

Development time and stability period

To test the effect of time on the absorbance of the coloured complex at the wavelength of maximum absorption at 542 nm, under the optimal experimental conditions. The absorbance was measured at different intervals of time. The experimental results shoed that the coloured complex develops immediately and the absorbance remains maximum and constant for at least 3 hr.

Beer's law, molar absorptivity and sensitivity

The linearity of the change in absorbance with variation in the amount of sulphite present was tested by reacting aliquots of the standard solution containing 0.1-150 µg of sulphite in a final volume of 25 ml and measuring the absorbance at 542 nm. Beer's law was obeyed over the range of 0.004-6 ppm of sulphite (Fig.2). The molar absorptivity being 4.64×10^4 $l.mol^{-1}.cm^{-1}$, and the sandell sensitivity is 0.001724 $\mu g\ .cm^{-2}$.

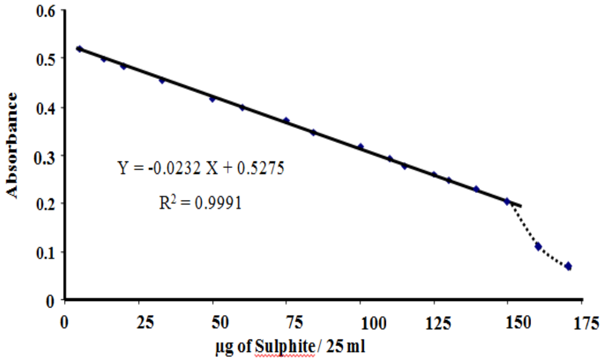


Figure 2. Calibration graph for Sulphite determination µg of Sulphite / 25 ml

Accuracy and precision

To check the accuracy and precision of the method, sulphite was determined at three concentrations. The results shown in Table 3 indicated that the proposed method was reliable.

Table 3. Accuracy and precision

Amount of Sulphite taken, µg/25ml	Recovery*, %	Relative standard deviation*, %
5	99.96	± 0.70
25	98.90	± 0.55
80	100.15	± 0.83

* Average of five determinations.

Nature of the reaction between chromate and (DPC) reagent.

Job's method (16) has been used in the determination of the reaction ratio of sulphite with chromate. The obtained results showed that 1:1 sulphite to chromate ratio is obtained. Also Job's method has been used in the determination of the reaction ratio of chromate with (DPC) reagent, the obtained results (Fig.3) showed that the ratio was 1:2 chromate to (DPC) reagent.

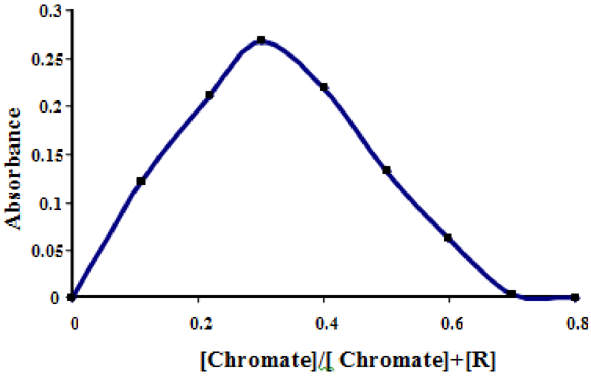


Figure 3. Job's plot for chromate – DPC

Also the stoichiometry of the reaction was investigated using the mole ratio method under the optimized conditions. The obtained results (Fig.4) showed that a 1:2 chromate to (DPC) reagent is obtained.

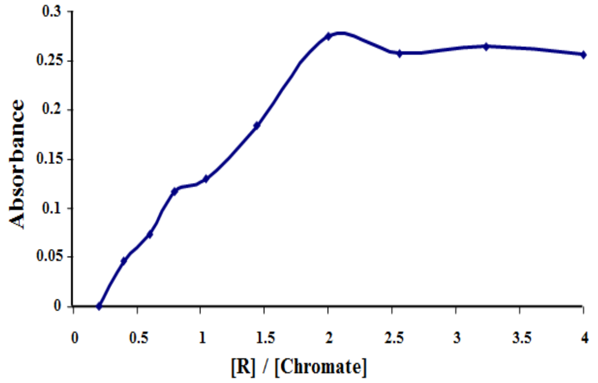
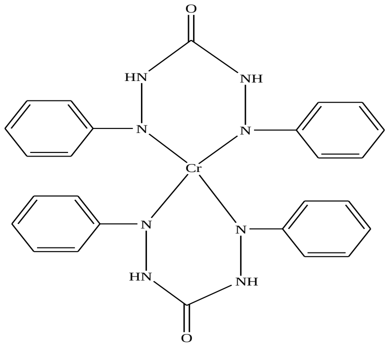


Figure 4. Mole ratio's plot for chromate -(PDC) reagent complex.

Hence the coloured complex may have the following structure:



Stability constant of formed complex

Stability constant of formed complex was calculated with ratio 1:2, different solutions contain equal quantities of chromate and reagent with (6.25×10^{-5}) M of each one. The absorbance for each sample was measured against its blank. The value of absorbance was expressed here by A_s. As also different solutions contain the same concentration of sulphite and excess of reagent's concentration. The value of absorbance was expressed here by A_m. The results are shown in Table 4, indicate that the average of stability constant is 2.8×10^{-12} $l.mol^{-2}$ which indicated that the colored complex was stable.

Table 4. Stability constant of formed coloured complex.

ml of 6.25×10^{-4} M chromate solution	Absorbance			$K.M^{-2}$
	A_s	A_m	α	
1.0	0.085	0.094	0.0957	4.88×10^{12}
2.0	0.129	0.223	0.4215	1.93×10^{12}
3.0	0.237	0.329	0.2796	1.63×10^{12}

Effect of foreign ions

The effect of various ions on the determination of sulphite was investigated. The results are shown in Table 5. It indicated that the method seemed to be moderately selective in the presence of composite masking solution, except towards $S_2O_3^=$, F^- and $C_2O_4^=$ which interfere seriously, also iodide in high concentrations.

Table 5. Effect of interferences on the determination of sulphite

Foreign ion	Form added	Relative error (%)			
		50	150	300	500
I^-	KI	+1.82	+2.56	+6.15	+9.33
F^-	NaF	-25.51	-27.33	-31.66	-35.76
Br^-	KBr	+4.78	+4.55	+6.15	+10.25
NO_2^-	$NaNO_2$	-1.28	-0.25	-0.24	-1.78
NO_3^-	$NaNO_3$	+1.66	+1.63	+0.99	+1.76
$CO_3^=$	Na_2CO_3	-1.67	-1.43	-1.32	-1.17
BO_3^{-3}	H_3BO_3	+0.19	+0.65	+0.34	+0.09
$SO_4^=$	Na_2SO_4	-0.29	-0.43	-0.59	-0.69
$S_2O_3^=$	$Na_2S_2O_3.5H_2O$	-74.82	-72.55	-70.03	-80.82
$PO_4^=$	$NaH_2PO_4.2H_2O$	+0.47	+0.58	+0.27	+0.63
$C_2O_4^=$	$Na_2C_2O_4$	-10.08	-14.58	-17.38	-19.27
CH_3COO^-	CH_3COONa	+0. 87	+0.91	+0.86	+0. 89
SCN^-	NaSCN	+2.81	+3.55	+4. 38	+4.81
Cd^{+2}	$Cd(CH_3COO)_2.H_2O$	+0.32	+0.21	+0.10	+0.42
Pb^{+2}	$Pb(NO_3)_2$	+0.83	+0.57	Turbid	Turbid
Bi^{+3}	$Bi(NO_3)_3.5H_2O$	-2.09	-1.27	-1.11	-2.90
Ba^{+2}	$BaCl_2.2H_2O$	+0.66	-0.62	-0.52	+0.78
NH_4^+	NH_4Cl	-0.49	-0.91	-0.52	-0.56
Fe^{+3}	$Fe(NO_3)_3.5H_2O$	+0.10	+0.82	+0.29	+0.20
Fe^{+2}	$FeSO_4.7H_2O$	+0.91	+2.82	+5.29	+7.20
Mg^{+2}	$Mg\ SO_4.7H_2O$	+0.26	-0.27	+0.63	+0.34
Hg^{+2}	$Hg(NO_3)_2.H_2O$	+0.98	+0.79	+0.41	+1.22
Th^{+4}	$Th(NO_3)_4.6H_2O$	+0.67	+0.63	+0.52	+0.87
Al^{+3}	$AlCl_3.6H_2O$	+0.22	+0.41	-0.64	+0.76
Cu^{+2}	$CuSO_4.5H_2O$	+1.19	+0.79	+0.52	+1.23

Determination of sulphite in presence of thiosulphate:

As thiosulphate ($S_2O_3^{-2}$) was clearly interfered, the proposed method have been developed for determining sulphite in presence of thiosulphate. This method depend on separating thiosulphate by precipitate as a complex of silver ions (17) using the following experiments:

Different volumes (0.5-2.5) ml of (0.01 M) silver nitrate solution were added to a solution containing a mixture of 10 µg sulphite and different amounts (50-500 µg) of thiosulphate then 2 ml of 1N nitric acid solution were added. After 2 min. a black precipitate was formed which then separated by filteration.

The filtrate was transferred to a 25 ml volumetric flask and the recommended procedure was applied for the determination of sulphite. The results given in Table (6) indicate that complete removal of thiosulphate (50 -500 µg) from sulphite was achieved using 2 ml of (0.01 M) silver nitrate solution as it given the nearest absorbance in comparison to the standered sulphite solution.

Table 6. determination of sulphite in presence of thiosulphate

ml of 0.01M $AgNO_3$	Absorbance */µg of $S_2O_3^{-2}$ present in 25 ml								
	50	75	100	150	200	250	300	400	500
0.5	0.408	0.418	0.426	0.432	0.438	Turbid	Turbid	Turbid	Turbid
1.0	0.443	0.438	0.446	0.453	0.459	0.422	0.418	Turbid	Turbid
1.5	0.455	0.458	0.466	0.462	0.449	0.463	0.461	0.459	0.464
2.0	0.493	0.493	0.499	0.495	0.497	0.490	0.504	0.502	0.493
2.5	0.511	0.519	0.507	0.519	0.517	0.521	0.518	0.511	0.513

* Absorbance without $S_2O_3^{-2}$ = 0.496

Application of the method

The method has been applied satisfactorily to the determination of sulphite in various water samples by standard comparison method. The results are compiled in Table 7.

Table 7. determination of sulphite in water samples

ml of water sample	Sulphite added µg	Recovery (%)				
		Tap water	Shalalat water*	Well water**	Natural spring water	Sea water***
1	5	101.09	99.87	99.34	100.12	100.09
3	5	103.18	99.74	99.64	100.15	101.08
5	5	101.49	99.67	99.91	100.25	100.29
7	5	100.19	100.17	100.21	99.91	101.17
1	50	99.73	101.55	100.19	99.83	99.93
3	50	98.92	99.28	99.83	99.84	98.82
5	50	99.97	99.49	99.23	99.80	99.87
7	50	100.18	100.42	100.51	98.91	100.15
1	100	101.29	100.22	99.80	100.09	101.19
3	100	98.99	100.13	100.29	100.01	99.93
5	100	99.83	99.34	100.20	99.85	99.79
7	100	99.66	99.89	98.97	99.94	99.26

Water samples applied from:-
* North of Mosul City, ** Al-Shoura village in Mosul, *** White middle sea in Syria .

Comparison of the method

Table 8 shows the comparison of spectrophotometric methods for sulphite determination.

Table 8.Comparison of the method

Analytical parameters	**Present method**	**Literature method (18)**
Method	Chromate-DPC	Fe(II)-1,10-Phenanthroline
pH	0.2	3.00
Buffer	-------	Glycine-HCl
λ_{max} (nm)	542	510
Reaction time (min)	2	Immediately
Stability period (minutes)	240	120
Beer's law range (ppm)	0.004-6.0	0.04-4.0
Molar absorptivity ($l.mol^{-1}.cm^{-1}$)	4.64×10^4	4.56×10^4
R.S.D. (%)	±0.55-±0.83	±0.52-±1.31
Colour of the product	Pink-violet	Orange -red
Application of the method	Water samples	Water samples

The present method seems to be rapide, sensitive, fair and can be applied to the determination of sulphite in various water samples.

The results in Table 9 show that the calculated value of t-test (19), did not exceed the theoretical values at the 95% confidence level for five degree of freedom indicating that there is no significant difference between the proposed method and the literature method.

Table 9.The value of "t" test of natural waters.

Sample	Tap water	Sea water	Tigris water	Natural spring water
The value of t-test	0.249	0.912	0.624	0.182

Conclusion:

A simple, sensitive, selective and inexpensive spectrophotometric method for the determination of sulphite in different water samples has been carried out by the rapid reduction of known amount of chromate CrO_4^{-2} in the presence of sulphite in acidic medium of sulphuric acid. Then the excess of chromate is measured when it reacts with 1,5-diphenylcarbazide as a reagent which finally gives a pink-violet, water soluble and stable complex, which exhibit maximum absorption at 542 nm. Beer's law were obeyed in the concentration range of 0.004-6.0 ppm of sulphite with a molar absorbtivity of 4.64×10^4 $L.mol^{-1}.cm^{-1}$, Sandell sensitivity index of 0.001724 $\mu g\ .cm^{-2}$ and relative standard deviation of ±0.55 - ±0.83 depending on the concentration level. The present method has been developed for the determination of sulphite in the presence of thiosulpate. Good recoveries of sulphite from various water samples are achieved using the proposed method.

Conflicts of Interest: None.

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التقدير الطيفي للكبريتيت في نماذج مائية مختلفة باستخدام معقد الكروم-5,1-ثنائي فنيل كاربازيد

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الخلاصة:

يتضمن البحث تقدير الكبريتيت بطريقة طيفية بسيطة وسريعة. وتعتمد الطريقة على اختزال الكرومات CrO_4^{-2} بوساطة الكبريتيت SO_3^{-2} في الوسط الحامضي باستخدام 2N من حامض الكبريتيك ومن ثم تقدير الكمية غير المختزلة من الكرومات بقياس طيف امتصاص المعقد الملون المتكون بين الكرومات غير المختزلة وبين الكاشف العضوي 1,5-ثنائي فنيل كاربازيد عند الطول الموجي 542 نانوميتر. كانت حدود قانون بير في مدى التركيز0.004-6.0 مايكروغرام من الكبريتيت في حجم نهائي 25 مللتر الامتصاصية المولارية 4.64×10^4 لتر.مول$^{-1}$.سم$^{-1}$. ودلالة ساندل للحساسية 0.001724 مايكروغرام.سم$^{-2}$ والانحراف القياسي النسبي في مدى ±0.55 إلى ±0.83 اعتمادا على مستوى التركيز. طورت الطريقة المقترحة لتقدير الكبريتيت بوجود الثايوكبريتات. وكانت للطريقة استرجاعية جيدة عند تطبيقها في تقدير الكبريتيت في نماذج مائية مختلفة.

الكلمات المفتاحية: الكبريتيت، طريقة طيفية، الكروم الثلاثي(III) كروات-DPC، 5,1-ثنائي فنيل كاربازيد.