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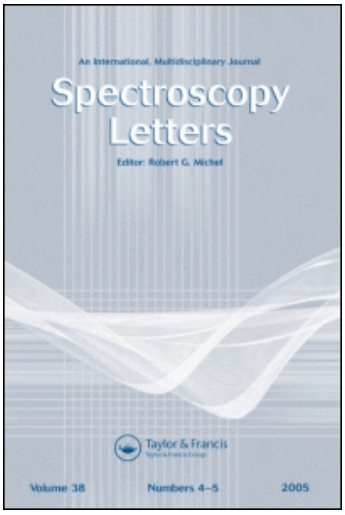

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Derivative Spectrophotometric and Colorbmetric Methods for the Determination of Guanoxan Sulphate in Tablets, Urine, and Serum.

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DERIVATIVE SPECTROPHOTOMETRIC AND COLORIMETRIC METHODS FOR THE DETERMINATION OF GUANOXAN SULPHATE IN TABLETS, URINE AND SERUM.

Keywords Derivative spectrophotometry; guanoxan sulphate; charge transfer complex; urine; serum.

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ABSTRACT:

Three simple and rapid methods for the determination of guanoxan sulphate in tablets, urine and serum are presented. The first method is based on the direct measurement of the first (^{1}D) and second (^{2}D) (peak-trough) derivative values at 274-

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250 nm and 276-260 nm respectively. The other two methods depend on the formation of a charge- transfer chromogen with either tetracyanoethylene (TCNE) and 7,7,8,8- tetracyanoquinodimethane (TCNQ). The absorbance of the color developed at 416 and 840 nm respectively. The first and second derivative (^1D & ^2D) values for the charge- transfer products were also measured. The methods were proved to be accurate and reproducible as indicated by relative standard deviation of less than 2%. The proposed methods have been applied to the determination of guanoxan sulphate in tablets, and spiked human urine and serum.

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INTRODUCTION:

Guanoxan sulphate {guanidine,[2,3-dihydro-1,4 benzodioxin-2-yl]methyl-sulphate} (Fig. 1) have been widely used as antihypertensive agent. It exhibit marked neuron blocking effects in the treatment of moderate and sever hypertension. Several methods have been reported for its determination in tablets or biological fluids, these include spectrophotometry[1], colorimetry[2], fluorimetry[3,4], high performance liquid chromatography[5] and gas chromatography after selective extraction procedure[6]. The reference method for the assay of guanoxan sulphate is based on the color - reaction of the guanidino group with sodium nitroprusside and potassium hexacanoferrate[7].

The conventional spectrophotometric analysis of drugs in dosage forms is often subjected to spectral interference from formulation matrix. The non specific irrelevant absorption may lead to serious systemic errors in the analytical growth curves of absorbance versus concentration. Such a problem becomes serious in the assay of

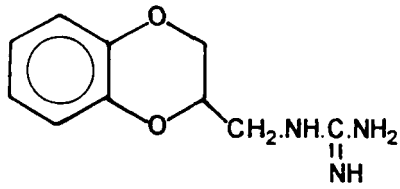

Fig. 1: Chemical structure of guanoxan.

tablets of weakly absorbing compounds which are formulated at relatively low dosage level (typically 1-50 mg/unit dose). The problem due to irrelevant absorption from formulation matrix has been treated by many mathematical methods including orthogonal function[8], least squares[9], and differential methods[10]. Such methods proved to be useful in certain cases but needed several steps of calculations and required a special attention in selecting the assay parameters. It has been shown that the application of derivative spectrophotometry offers an elegant and simple approach for resolving spectral overlap and for quantitation of drugs in dosage forms[11].

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π- Acceptors such as tetracyanoethylene (TCNE) and 7,7,8,8-tetracyanoquinodimethane (TCNQ) are known to yield charge-transfer complexes and radical anions with a variety of electron donors. TCNE and TCNQ have been used for the determination of several compounds including amines[12], phenols, aromatic hydrocarbons[13] and alkaloids[14].

In this work, three spectrophotometric methods for the determination of guanoxan sulphate are described. The first method is based on the measurement of the

derivative (^1D & ^2D) values of the zero order of the spectrophotometric curve of guanoxan sulphate. While the other two methods depend on the formation of a charge-transfer product between guanoxan sulphate and TCNE or TCNQ with sharp maxima at 416 and 840 nm, respectively. Furthermore, the derivative (^1D & ^2D) values for the previous charge-transfer products were measured at the specified wavelengths, to increase the sensitivity of the method. The above proposed spectrophotometric methods were successfully applied to the determination of guanoxan sulphate in spiked human urine and serum after selective extraction.

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EXPERIMENTAL

Apparatus

A Perkin-Elmer Model 550S UV-visible spectrophotometer with 1-cm quartz cuvettes and a Hitachi Model 561 recorder were used. The following operating conditions were used: scan speed 120 mm/min, chart speed 60 nm/min; $^1D = (dA/d\lambda)$, $^2D = (d^2A/d\lambda^2)$. The minimum and maximum ordinate settings were selected to record the maximum amplitude of the most concentrated standard solution at not less than 80% of the recorder full scale deflection (25 cm).

Materials and reagents

All materials and reagents were of analytical grade. A pharmaceutical grade of guanoxan sulphate was kindly provided by (Pfizer, Kent, UK.). Tablets containing guanoxan sulphate were prepared in laboratory to contain 10 mg guanoxan sulphate

per tablet. The fillers and excipients used were loctose 90, starch 7, talc 2.7 and magnesium stearate 0.3 parts. Tetracyanoethylene (TCNE) and 7,7,8,8-tetracyano-quinodimethane (TCNQ) obtained from Aldrich Chem. Co. and solutions prepared to contain 0.1% from the former and 0.2% from the latter in acetonitrile. Biological samples, serum and urine specimens were collected from adult healthy volunteers who were not under medical treatment.

Derivative method:

(1) Preparation of calibration graphs

A 20 mg aliquot of the drug was accurately weighed and dissolved in 100 ml of distilled water. Working standard solutions were prepared by dilution of stock solution with 0.1 M hydrochloric acid to reach concentration range stated in Table 1. The first derivative (^1D) and second derivative (^2D) of the zero order U.V. spectrum of guanoxan sulphate were recorded against O.1 M Hydrochloric acid using the above mentioned instrumental parameters. The peak-trough values for ^1D and ^2D of the derivative curves (Fig. 2) were then measured at the chosen wavelengths (Table 1).

(2) Tablet assay

Accurate weight of the powdered tablets equivalent to 20 mg of guanoxan sulphate was extracted by shaking for 20 min with distilled water (70 ml). The extracts were filtered through decantation on filter paper into 100 ml volumetric flask

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TABLE 1

Analytical Data of the Calibration Graphs for the Determination of Guanoxan Sulphate by the Proposed Methods.

Method	Selected Wavelength λ (nm)	Concentration range ug/ml	Linear regression Intercept (a)	Slope (b)	Corr. coff. (r)	$S_{y/x}$ +	S_a ++	S_b +++	RSD* (%)	Apparent molar absorptivity ∈ (1 mol^{-1} cm^{-1})
^1D	274,250	1-7	-0.005	2.620	0.9998	0.154	0.167	0.03	1.10	-
^2D	276,260	1-7	0.129	3.042	0.9997	0.141	0.153	0.03	1.20	-
TCNE										
^1D	418	2-10	0.157	1.43	0.9995	0.152	0.154	0.024	1.30	$2.96 X10^{-3}$
^2D	422,404	2-10	-0.200	1.86	0.9999	0.111	0.113	0.017	0.83	$3.85 X10^{-3}$
TCNQ										
^1D	844	3-10	0.057	0.88	0.9996	0.087	0.096	0.015	1.50	$1.82 X10^{-3}$
^2D	848,832	3-10	0.310	1.77	0.9998	0.112	0.124	0.020	1.00	$3.67 X10^{-3}$

\+ $S_{y/x}$ = standard deviation of residuals (n=6)
++ S_a = standard deviation of intercept of regression line
+++ S_b = standard deviation of slope of regression line
* Relative standard deviation (n=5)

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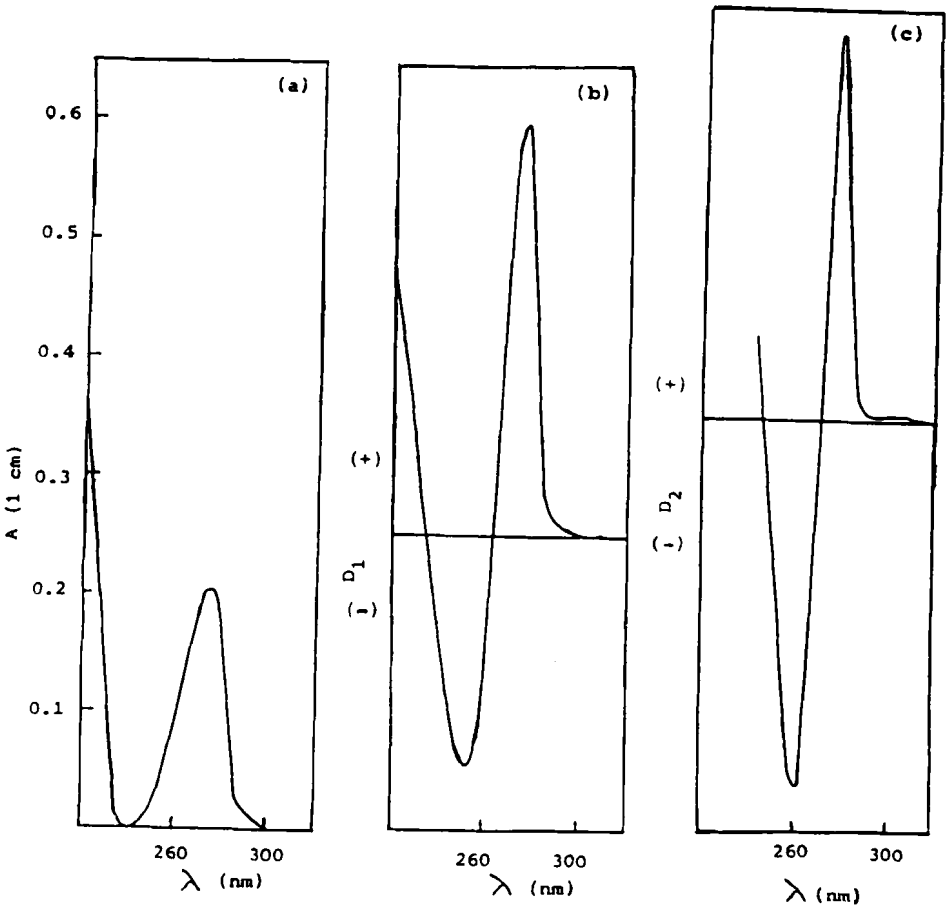

Fig. 2: (a) Absorption spectra (—) of 20 ug/ml guanoxan sulphate and its (b) first (——) and (c) second (——) derivative spectra of 5 ug/ml, in 0.1 N hydrochloric acid.

and the volume was made up using distilled water. The procedure was completed as under preparation of calibration graphs. The concentration of the drug in the sample was determined by reference to the appropriate calibration graph.

Charge transfer methods:

(1) Standard drug solution

Accurate weight of guanoxan sulphate equivalent to 25 mg was transferred into a 100 ml separatory funnel and dissolved in about 20 ml distilled water. The solution was rendered alkaline with few drops of 10% sodium hydroxide and extracted successively with three 25-ml portions of chloroform. Each chloroform extract was filtered through a filter paper containing anhydrous sodium sulphate. The chloroform extracts were collected in a 100 ml volumetric flask and diluted to volume with chloroform. The drug base solution was further diluted with chloroform to give a solution containing 0.1 mg/ml of the analyte.

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(2) Preparation of calibration graphs

Accurate volumes of standard drug base solution (0.1 mg/ml) within the concentration range stated in Table 1 were transferred into separate 10 ml volumetric flasks. The solvent was removed by immersing the flasks in a water bath at 70°C till dryness then cooled. A 1.0 ml TNCE solution or 1.5 ml TNCQ solution was added to each flask and completed to volume with acetonitrile.

For TNCE-method

The ^1D and ^2D spectra were recorded against similarly prepared blank. The ^1D-values at 418 nm (Peak height) and ^2D-values at 422-404 nm (Peak-trough) were measured.

For TCNQ-method

The solutions were left at room temperature for 45 min and The ^1D and ^2D spectra were recorded against similarly prepared blank. The peak height values were measured at 844 nm for ^1D spectrum and peak-trough values were measured at 848-832 nm for ^2D spectrum.

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(3) Tablet assay

Accurate weight of the powdered tablets equivalent to 40 mg of guanoxan sulphate were extracted with three 25 ml portions of water. The extracts were filtered through decantation on filter paper into a 100-ml volumetric flask. Twenty five ml portion of this solution was transferred into 100-ml separatory funnel. The drug base was extracted as under preparation of standared drug solution starting from "The solution was rendered.....". Then the procedure was completed as under preparation of calibration graphs replacing standard solution by tablet solution for TNCE and TCNQ methods.

Determination of guanoxan sulphate in spiked human urine and serum using derivative spectrophotometric method and charge transfer methods:

The pH of urine were adjusted to 10.0 and to 7.0 for serum. 5-ml aliquot of urine or serum were placed in 50-ml stoppered shaking tubes. Fourty mg of guanoxan sulphate was accurately weighed and transfered to stoppered shaking tube containing urine or serum. Twenty ml toluene in case of urine or 20-ml diethyl ether in case of serum were added, the contents were extracted by shaking for 10 min, then centrifuged. The organic layer were rejected. 0.5 ml 50% sodium hydroxide were added and extracted for 20 min using 30 ml dichloromethan. The organic layer were transferred to anthor shaking tube containing 10-ml 0.1 N hydrochloric acid, the content were extracted for 10 min and then centrifuged. The aqueous layer was transferred to 100-ml volumetric flask and completed to volume with distilled water.

- For derivative method, 25-ml portion of the extracted solution was transferred to a 100-ml volumetric flask and completed to volume with 0.1 N hydrochloric acid. The procedure was completed as under preparation of calibration graph. The concentration of drug in urine or serum was determined by reference to the appropriate calibration graph.
- For charge transfer methods, 25 ml portion of the extracted solution was transferred into a 100-ml separatory funnel. The drug base was extracted as under preparation of standared drug solution starting from "The solution was rendered....". Then the procedure was completed as under preparation of calibration graphs. The concentration of drug in urine or serum was determined by reference to the appropriate calibration graphs.

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RESULTS AND DISCUSSION

Derivative method

Guanoxan sulphate is presented in low dose (10 mg per tablet). In addition, this drug is weakly absorbing [Fig. 2(a)] in the ultraviolet region [with $A^{1\%} = 104$[15]]. Therefore, the low absorbance and high excipient-drug ratio prohibit the application of the conventional spectrophotometric method. Meanwhile, the first and second-derivative techniques can correct for the interference due to excipients.

Figure 2(b,c) shows first (^1D) and second (^2D) derivative spectra of guanoxan sulphate which exhibit characteristic peaks at certain wavelengths. Therefore, the peak-trough measurements for the ^1D and ^2D spectra at the chosen wavelength were selected for its determination (Table 1).

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Charge - transffer method

Guanoxan, being n-electron doner react instantaneously with the π- acceptors TNCE and TNCQ in acetonitrile to give charge-transfer complexes and radical anions[12,13]. The maximum absorbance bands are at 416 nm (yellow color) and at 840 nm (green color) for TNCE and TNCQ reactions, respectively [Fig. 3,4(a)].

The reaction conditions for the two method have been optimized; for each reagent concentration, reaction time and stability of the chromogen with respect to maximum sensitivity and obdience to Beer's law [Fig. 5,6]. Under the given experimental

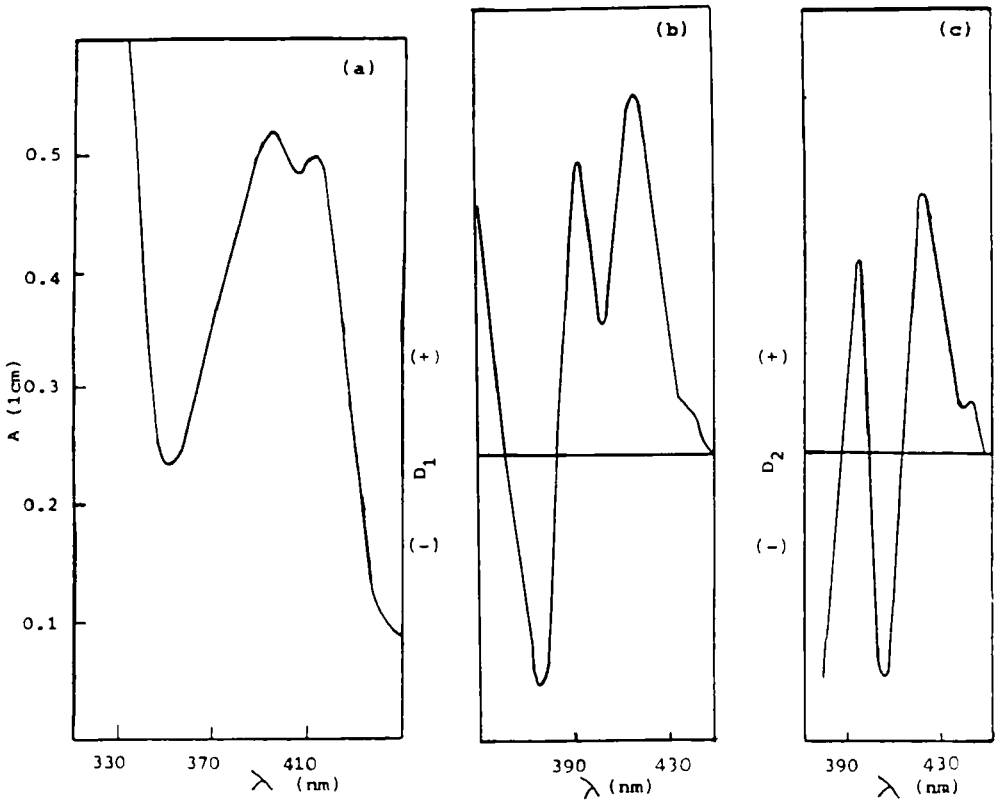

Fig. 3: (a) Absorption spectra (—) of 15 ug/ml guanoxan- TCNE complex and its (b) first (——) and (c) second (——) derivative spectra of 5 ug/ml in acetonitrile.

conditions, the absorbance of the colors developed was stable for at least one hour for both reagents.

In order to increase the sensitivity of the charge-transfer methods, the first and second derivative techniques were applied for measuring guanoxan- TNCE complex and guanoxan-TCNQ complex. Figures 3,4(b,c), represent the first and second

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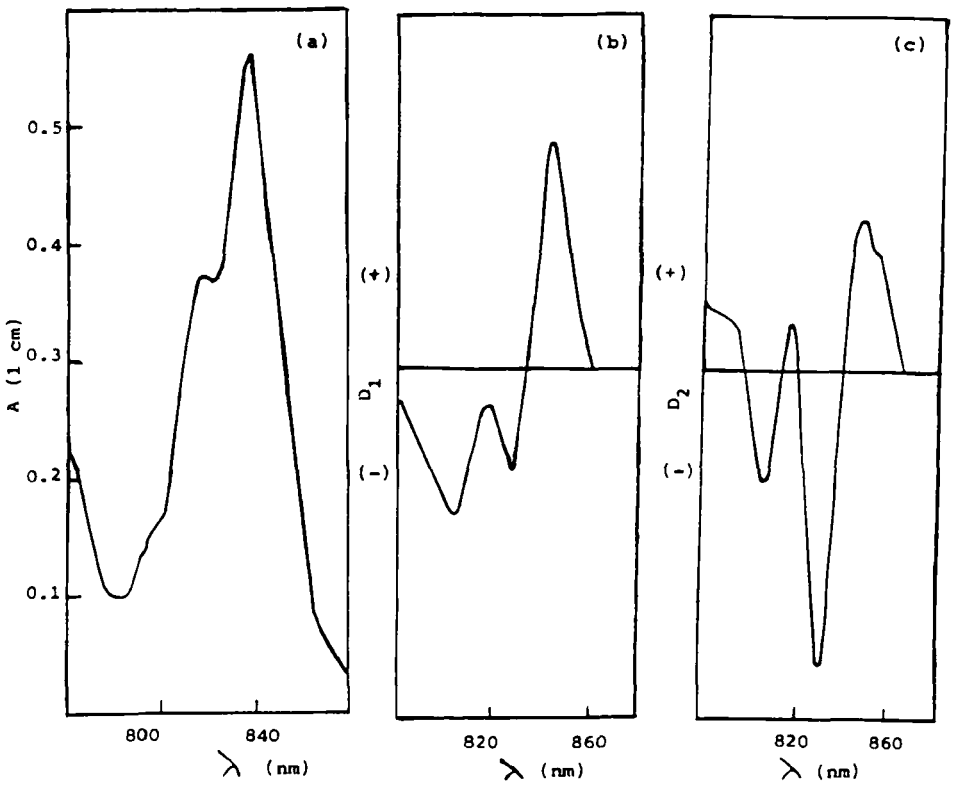

Fig. 4: (a) Absorption spectra (——) of 15 ug/ml guanoxan-TCNQ complex and its (b) first (——) and (c) second (——) derivative spectra of 5 ug/ml in acetonitrile.

derivative spectra of 5 ug/ml guanoxan- TCNE complex and guanoxan- TCNQ complex respectively. The measurements of derivative values at specified wavelengths (Table 1) of the two charge transfer complexes enhance the sensitivity of the method to be three times more sensitive than direct absorbance measurement.

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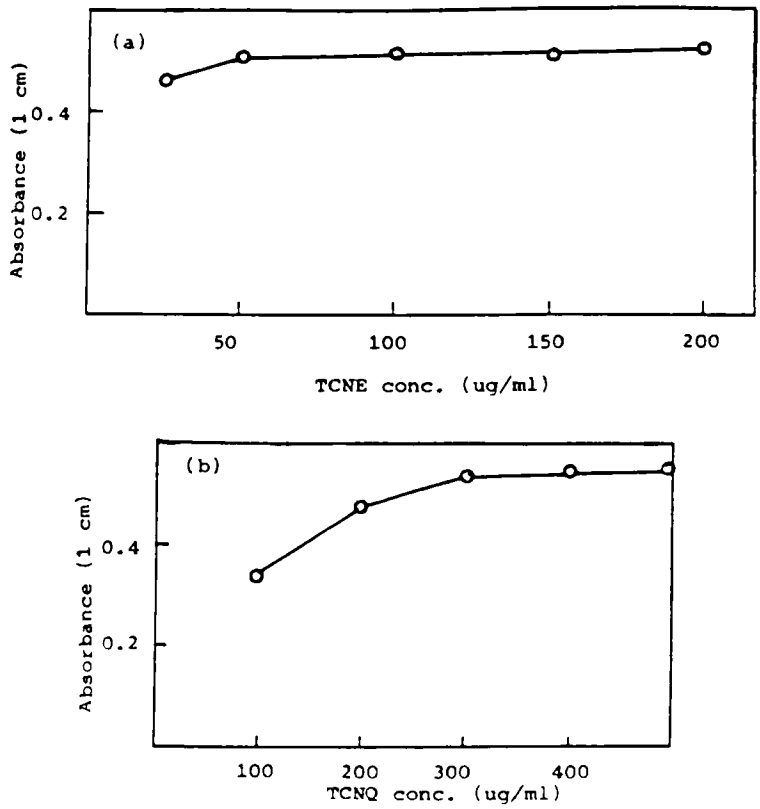

Fig. 5: Effect of (a) TCNE (—o—) (b)TCNQ (—o—) concentrations on the charge-transfer complex developed from their reaction with 15 ug/ml guanoxan.

Linearity, sensitivity and selectivity:

The calibration graphs were constructed for the proposed methods from six data points over the concentration range cited in Table 1. The latter presents the results of the statistical analysis of the experimental data, the regression equations calculated from the calibration graph, along with the standard deviation of the slope (S_b) and the intercept (S_a) on the ordinate and the standard deviation of residuals ($S_{y/x}$). The

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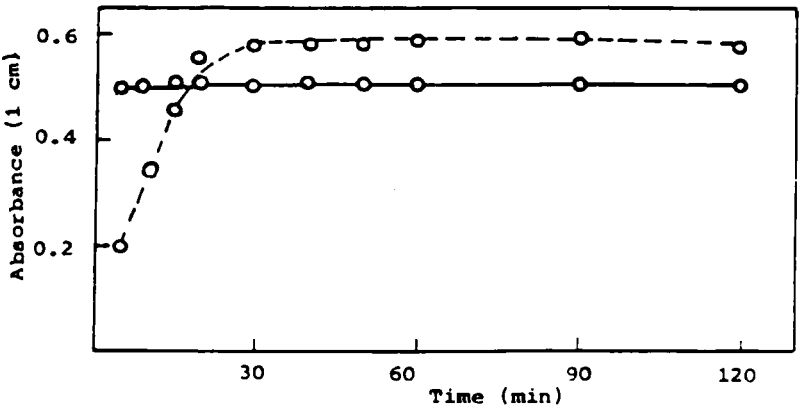

Fig. 6: Effect of reaction time on the charge-transfer complex developed from the reaction of TCNE (—) and TCNQ (------) with 15 ug/ml guanoxan.

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linearity of the calibration graphs and conformity of the ^{1}D, ^{2}D measurements of the proposed methods to Beer's law are proved by the high values of the correlation coefficients (r) of the regression equations.

Five replicate determination at different concentration levels were carried out to test the precision and reproducibility of the proposed methods. The relative standard deviations were found to be less than 2% indicating reasonable repetability of the methods (Table 1). the apparent molar absorptivities for the charge transfer method (TNCE and TCNQ) are presented in Table 1. The detection and a quantification limits[16,17] were calculated for the guanoxan sulphate using the different proposed methods (Table 2).

TABLE 2

The Detection and Quantification Limits of Guanoxan Sulphate Using The Proposed Methods.

Proposed method	C_L^+ ug ml^{-1}	C_Q^{++} ug ml^{-1}
$^1D_{274.250}$	0.068	0.226
$^2D_{276.260}$	0.058	0.193
TCNE		
$^1D_{418}$	0.314	0.150
$^2D_{404.422}$	0.242	0.806
TCNQ		
$^1D_{844}$	0.68	1.55
$^2D_{832.848}$	0.34	1.13

\+ $C_L = 3S_B/b$; detection limit, S_B standard deviation of blank; b = slope of calibration graph.

++ $C_Q = 10\ S_B/b$; quantification limit.

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Analysis of tablets

The proposed methods were evaluated in the assay of the laboratory prepared tablets (prepared to contain 10 mg guanoxan sulphate per tablet). Five replicate determination were carried out on an accurately weighed amount of pulverised tablets, giving excelent % recovery (Table 3) with percentage relative standard deviation (RSD%) less than 1.0% and percentage relative error (Er%) less than 0.52%. There results conform satisfactory to the lable claim and indicate the high precision and accuracy of the proposed methods when applied to tablets.

TABLE 3

Assay Results of Guanoxan Sulphate Tablets* Using the Proposed Methods and the Reference Method.

Method n = 5	Recovery ± standard deviation** (%)	t	F	E_r (%)***
^{1}D	100.5 ± 0.74	0.64	1.03	0.520
^{2}D	100.4 ± 1.00	0.36	1.83	0.480
TCNE				
^{1}D	99.5 ± 0.31	1.94	5.85	0.512
^{2}D	99.6 ± 0.36	1.60	4.34	0.521
TNCQ				
^{1}D	99.8 ± 0.61	0.93	1.51	0.211
^{2}D	99.8 ± 0.58	0.94	1.67	0.231
Reference	100.2 ± 0.75			

* Laboratory made tablets: prepared to contain 10 mg guanoxan sulphate per tablet.
** Average of n replicates. The theoretical t-value (p=0.05) is 2.31. The theoretical F-value (95%) is 6.39.
*** Percentage relative error.

The performance of the proposed methods was statistically compared with that of the reference method by student's t-test and variance ratio F-(Table 3). The calculated t-and F-values did not exceed the theorotical values in either test, indicating that there were no significant difference between the proposed methods and reference method.

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In vitro determination of guanoxan sulphate in spiked human urine and serum using the proposed methods.

The proposed methods were applied for the in vitro determination of guanoxan sulphate in urine and serum after selective extraction[6]. Within-day precision (random analytical variation) was evaluated by replicate analysis of urine and serum samples containing guanoxan sulphate at concentration range cited in Tables 4,5. Day- to Day precision (total analytical variation) was similary evaluated on several days over two weeks and no more than one assay per day for each concentration. The data presented in Tables 4,5 indicate that the 24 h and day- to day coefficents of variation are generally lower than 1.0%.

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Absolute recovery was studied by adding known amounts of guanoxan sulphate to drug- free urine and serum at different concentrations. Five determinations were performed for each concentration. Data obtained were compared to that obtained by using standard guanoxan sulphate solution. The results are presented in Table 6.

The relative recovery was calculated by comparing the concentrations obtained from the drug-supplemented urine and serum with standard solution treated in the same manner. The results shown in Table 6, indicate good rproducibility and accuracy of the proposed methods.

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TABLE 4

Within-Day and Day-to-Day Precision and Relative Recovery in the Detemination of Guanoxan Sulphate in Spiked Human Urine.

Method	Derivative Spectrophotometry % Recovery* (C.V. %)**				TCNE % Recovery* (C.V. %)**				TNCQ % Recovery* (C.V. %)**			
Add ug/ml	Within day (n=6)		Day-to-Day (n=6)		Add ug/ml	Within day (n=6)		Day-to-Day (n=6)		Add ug/ml	Within day (n=6)		Day-to-Day (n=6)	
	^1D	^2D	^1D	^2D		^1D	^2D	^1D	^2D		^1D	^2D	^1D	^2D
2	99.7(0.21)	99.5(0.31)	100.7(0.17)	100.3(0.20)	4	101.7(0.09)	101.1(0.13)	101.2(0.16)	101.3(0.09)	4	100.2(0.12)	100.5(0.07)	100.7(0.17)	100.6(0.08)
4	101.2(0.15)	100.9(0.12)	100.9(0.10)	101.0(0.07)	6	100.6(0.11)	100.9(0.32)	100.6(0.19)	100.2(0.16)	6	99.3(0.13)	100.0(0.10)	101.1(0.09)	100.9(0.13)
6	99.6(0.11)	100.0(0.20)	98.1(0.21)	99.0(0.11)	8	99.9(0.26)	100.3(0.21)	100.3(0.11)	100.7(0.22)	8	99.6(0.11)	99.2(0.05)	99.7(0.11)	100.0(0.05)

* Mean of five experiments.

** Cofficient of variation.

TABLE 5

Within-Day and Day-to-Day Precision and Relative Recovery in the Determination of Guanoxan Sulphate in Spiked Human Serum.

Method	Derivative Spectrophotometry % Recovery* (C.V. %)**				TCNE % Recovery* (C.V. %)**				TNCQ % Recovery* (C.V. %)**			
Add ug/ml	Within day (n=6)		Day-to-Day (n=6)		Add ug/ml	Within day (n=6)		Day-to-Day (n=6)		Add ug/ml	Within day (n=6)		Day-to-Day (n=6)	
	^1D	^2D	^1D	^2D		^1D	^2D	^1D	^2D		^1D	^2D	^1D	^2D
2	98.9(0.18)	98.4(0.13)	99.3(0.18)	99.0(0.14)	4	98.4(0.05)	98.6(0.09)	99.4(0.14)	99.7(0.16)	4	99.9(0.15)	100.3(0.09)	100.0(0.13)	100.4(0.10)
4	99.3(0.11)	99.7(0.19)	99.8(0.09)	100.0(0.05)	6	98.9(0.12)	99.3(0.13)	99.9(0.02)	100.3(0.08)	6	98.4(0.21)	99.1(0.11)	99.5(0.17)	99.1(0.10)
6	99.9(0.14)	99.6(0.08)	100.1(0.17)	100.0(0.05)	8	100.0(0.08)	100.1(0.17)	100.1(0.11)	100.3(0.17)	8	100.3(0.09)	100.4(0.10)	100.4(0.18)	100.0(0.05)

* Mean of five experiments.

** Cofficient of variation.

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TABLE 6

Analytical Recoveryof Guanoxan Sulphate from Spiked Human Urine and Serum Using the Proposed Methods.

Method	Spiked Urine		Spiked Serum	
	Absolute recovery % (mean* ± S.D.)	Relative recovery % (mean* ± S.D.)	Absolute recovery % (mean* ± S.D.)	Relative recovery % (mean* ± S.D.)
^{1}D	89.15 ± 3.81	100.16 ± 0.89	85.4 ± 3.11	99.2 ± 0.55
^{2}D	90.30 ± 4.01	100.13 ± 0.70	87.4 ± 4.23	99.2 ± 0.72
TCNE				
^{1}D	86.3 ± 3.21	100.7 ± 0.91	90.1 ± 3.12	99.1 ± 0.82
^{2}D	88.4 ± 5.58	100.8 ± 0.42	92.4 ± 6.11	99.3 ± 0.75
TCNQ				
^{1}D	91.3 ± 6.06	99.7 ± 0.46	87.0 ± 6.21	99.5 ± 1.00
^{2}D	98.9 ± 5.45	99.9 ± 0.66	89.4 ± 5.88	99.9 ± 0.72

* Mean of five experiments.

CONCLUSION

The proposed methods is recomended for the routine analysis of guanoxan sulphate in tablets. Direct derivative methods was found to be simple, selective and rapid. The application of the derivative technique to the measurements of the charge-transfer complexes (TNCE and TCNQ) has greatly increase the sensitivity of these two methods.

Therefore the proposed methods were successfuly applied for the determination of guanoxan sulphate in tablets and also in spiked human urine and serum without any interference.

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