

## SPECTROPHOTOMETRIC DETERMINATION OF DAPSONE FROM PHARMACEUTICAL PREPARATION

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### ABSTRACT

A simple diazotisation technique and its subsequent coupling with three different reagents have been successfully employed in spectrophotometric determination of dapsone from pharmaceutical preparations. The diazotisation was effected with a mixture of trichloroacetic acid and sulphuric acid. The method was found to be more sensitive than those reported in the literature.

### INTRODUCTION

DAPSONE, 4,4'-sulphonyldianiline, is used as an antibacterial (leprostatic) and is incorporated commonly as plain dapsone tablets. An assay based on titration of dapsone with 0.1 M sodium nitrite solution has earlier been described<sup>1-3</sup>. While two methods describe the potentiometric determination of the end point, the third determines the end point by blue colour with starch-iodide paper. A colorimetric method<sup>4</sup> to estimate dapsone from blood serum and a gas chromatographic method<sup>5</sup> to analyse dapsone have also been reported where a U-shape glass column packed with 3% poly-A-103 on Gas-chrom QC(100 to 120 mesh) operated at 285°, with nitrogen as carrier gas (100 ml/min) is used. Electron capture detector with a <sup>63</sup>Ni source was used. Yet another method to estimate dapsone from plasma has been suggested.<sup>6</sup> Metol reagent to determine dapsone colorimetrically at 520 nm has also been proposed.<sup>7</sup>

A spectrophotometric method reported here is based on the coupling of diazotised dapsone with oxine, R-salt of  $\beta$ -naphthol and 8-amino-1-naphthol 3:6 disulphonic acid (Na salt) in an acid mixture of trichloro acetic acid and sulphuric acid.

### EXPERIMENTAL

#### *Instrument*

CZ spekol spectrophotometer with 1 cm matched glass cells.

#### *Reagents and solutions*

- (A) (a) *Acid mixture*: A mixture of 86 ml of 15% trichloroacetic acid, 20 ml of 4 N sulphuric acid was diluted to 200 ml with distilled water.  
 (b) *Sodium nitrite*: 1 mg/ml solution was prepared in distilled water.  
 (c) *Ethyl alcohol*: 95%.  
 (d) *Trimethylamine*: 60% strength.  
 (e) *Coupling reagents*:

- (i) Oxine: 2mg/ml in 50% of absolute alcohol.  
 (ii) R-salt of  $\beta$ -naphthol: 1 mg/ml in distilled water.  
 (iii) 8-amino 1-naphthol. 3 : 6 disulphonic acid (Na-salt): 1 mg/ml in distilled water.  
 (B) Standard stock solution of dapsone of 1 mg/ml strength was prepared in alcohol. 10 ml of the standard stock solution was further diluted to 50 ml in water to give 200  $\mu$ g/ml working standard solution.

### PROCEDURE

#### *Method A: Determination of dapsone with oxine and R-salt of $\beta$ -naphthol*

In a series of 25 ml standard volumetric flasks, 0.1 to 0.7 ml aliquots of working standard solution were pipetted separately. To each flask, acid mixture and 0.5 ml of NaNO<sub>2</sub> were added. The solutions were allowed to stand for 5 minutes and the coupling reagents were then added to each flask with shaking and the colour was developed by adding trimethylamine. The flasks were allowed to stand for 10 minutes for colour development. After dilution to the mark with distilled water, the absorbance values were measured at the respective  $\lambda_{\max}$  against the reagent blank. The Beer's law plot of absorbance against concentration in  $\mu$ g/ml was plotted. (The amount of acid mixture, coupling reagent and trimethylamine required is given in table 1).

#### *Method B : Determination with 8-amino-1-naphthol 3 : 6 disulphonic acid (Na-salt)*

In a series of 25 ml standard volumetric flasks, 0.1 to 0.6 ml aliquots of working standard solution were pipetted separately. To each flask 1 ml of acid mixture and 0.5 ml of NaNO<sub>2</sub> were added. The solutions were allowed to stand for 5 min. and 5 ml of ethyl alcohol was then added to each flask. The colour was developed by adding 3 ml of coupling reagent to each flask. The flasks were allowed to stand for 10 minutes for colour development. After dilution to the mark with distilled water, the absorbance values were

TABLE I  
Results of determination of dapsone

Reagent	Beer's law range $\mu\text{g/ml}$	Stability of colour in min	$\lambda_{\text{max}}$ nm	Acid mixture used (ml)	Coupling reagent used (ml)	Tri-methylamine used (ml)	% recovery	S.D.	Coefficient of variation %	Molar absorptivity litre mole <sup>-1</sup> cm <sup>-1</sup> $\times 10^4$
Oxine	0.8-5.6	10-30	505	1	2	4	101.03	0.04	1.51	3.72
R-salt of $\beta$ -naphthol	0.8-5.6	10-40	500	2	4	3	97.14	0.07	1.63	3.30
8-amino-1-naphthol 3 : 6 disulphonic acid (Na-salt)	0.8-4.8	10-40	535	1	3	—	100.83	0.03	0.95	3.97

measured at 535 nm against the reagent blank. The Beer's law plot of absorbance against concentration in  $\mu\text{g/ml}$  was plotted.

#### APPLICATION TO PHARMACEUTICAL PREPARATIONS

Twenty tablets of dapsone were accurately weighed and powdered. Powder equivalent to 100 mg of dapsone was dissolved in 100 ml of alcohol and thoroughly shaken. The solution was filtered and 10 ml of the sample solution was diluted to 50 ml in a standard flask with distilled water to give a working sample solution of 200  $\mu\text{g/ml}$ .

Colour development was carried out as detailed earlier and the drug present in the formulation was computed from calibration curve.

#### RECOVERY EXPERIMENT

To study % recovery and accuracy of the proposed method, statistical studies were carried out. A fixed amount of the sample was taken in 25 ml standard volumetric flasks and three different levels of standard solutions were added. Each level of the added drug was repeated seven times. The total amount of the drug was then determined by the proposed method. The % recovery was calculated by using the equation:

$$\% \text{ recovery} = \frac{N \sum XY - (\sum X)(\sum Y)}{N \sum X^2 - (\sum x)^2} \times 100$$

where  $N$  = total number of observations,  $X$  = amount of the drug added and  $Y$  = amount of the drug found by the proposed method.

The results of determination of dapsone by all three reagents are given in table I.

#### DISCUSSION

Assay procedures to determine dapsone by nitrite titration have earlier been described<sup>1-3</sup>. However, these methods require milligram quantities of the drug. The present method requires only microgram quantities of the drug. The method does not also suffer interference from common excipients like starch, talc and other binding material. The other colorimetric methods described in the literature require the addition of ammonium sulphamate to remove excess nitrite ions which interfere in the coupling reaction. The present method utilizes an acid mixture consisting trichloroacetic acid and sulphuric acid whereby excess nitrite ions are destroyed and the reaction carried out at room temperature.

The colour development with all the proposed reagents is instantaneous and the colour is sufficiently stable. The results of % recovery, standard deviation and coefficient of variation indicate that the proposed methods are accurate and precise. The molar absorptivity values clearly show that the proposed method is more sensitive than similar methods reported in the literature.

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## SOLVENT EXTRACTION AND SPECTROPHOTOMETRIC DETERMINATION OF VANADIUM (V) USING N-HYDROXY-N-m-TOLYL-N'-PHENYLBENZAMIDINE

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### ABSTRACT

A method has been devised for extractive separation and simultaneous spectrophotometric determination of vanadium (V) employing N-hydroxy-N-m-tolyl-N'-phenylbenzamidine (HTPB). The method is based on the formation of a water insoluble blue-violet vanadium (V) complex in 1.0 to 10.0 M acetic acid, which can be extracted with chloroform. The complex shows a broad absorption maxima at 560–580 nm, when measured against chloroform blank. The  $\lambda_{\max}$  (570 nm) of the complex and that of the reagent (313 nm) are well separated, hence the excess of the reagent does not interfere in the photometric determination. The method is rapid, sensitive and highly selective. The separation and determination of vanadium (V) can be carried out in the presence of a large number of foreign ions including Fe(III), Mo(VI), Cr(III), Mn(II) and Ni(II). The method has been successfully applied to determine the vanadium content of BCS steels accurately.

### INTRODUCTION

N-BENZOYL-N-phenylhydroxylamine (BPHA) and its analogues<sup>1-4</sup> have been recommended for determination of vanadium (V) but lack selectivity. Some investigators<sup>5,6</sup> have pointed out that in this method the extraction is not quantitative because of partial reduction of vanadium in concentrated hydrochloric acid. In the present communication, N-hydroxy-N-m-tolyl-N'-phenylbenzamidine has been found to be a sensitive and highly selective reagent for the extractive separation and photometric determination of trace quantities of vanadium. The drawbacks mentioned above have been successfully overcome by this method. No partial reduction takes place, hence extraction is quantitative. The method has been applied to determine the vanadium content of BCS alloys.

### EXPERIMENTAL

Standard vanadium (V) solution was prepared by dissolving A.R. ammonium metavanadate in double distilled water and standardized titrimetrically<sup>7</sup>. An ECIL spectrophotometer (UV/VIS) model GS-865 was used.

N-Hydroxy-N-m-tolyl-N'-phenylbenzamidine was prepared by the method of Deb and Mishra<sup>8</sup>. The resulting compound was crystallized from benzene. M.P. 138°; yield 52%; Elemental analysis: (Found: C, 79.60%; H, 6.10%; N, 9.35%; Calculated for C<sub>20</sub>H<sub>18</sub>N<sub>2</sub>O; C, 79.47%; H, 5.95%, N, 9.27%). A, 0.1% solution of the reagent in chloroform was used for extraction purposes.

### *Recommended procedure*

An aliquot of the solution containing 100  $\mu$ g of vanadium was taken in 60 ml separatory funnel and to it, 10 ml of glacial acetic acid and sufficient distilled water was added to adjust the volume of aqueous phase to 25 ml, 6 ml of 0.1% solution of the HTPB reagent in chloroform was then introduced. The contents were shaken vigorously for 1 min and the chloroform layer was separated and dried with 2 g of anhydrous sodium sulphate. The aqueous layer was washed twice with 4 ml of chloroform and the combined chloroform extract dried over anhydrous sodium sulphate, and made up the volume to 25 cc and measure the absorbance at 570 nm against chloroform blank.