



**SPECTROPHOTOMETRIC METHODS FOR THE DETERMINATION OF
DEMECLOCYCLINE, DOXYCYCLINE AND HOSTACYCLINE**

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ABSTRACT

New, rapid and sensitive spectrophotometric methods have been developed for the determination of Demeclocycline(DMC), Doxycycline(DC) and Hostacycline(HTC). The procedure is based on the observation that, DMC, DC and HTC forms coloured complex with vanadium(V), the absorbance of which is proportional to the amount of drug present. The variable affecting development of the colour have been investigated and the conditions are optimized.

KEYWORDS: Spectrophotometric determination, Demeclocycline, Doxycycline and Hostacycline, vanadium(V).

1. INTRODUCTION

Hostacycline (HTC) is a bright yellow crystalline salt. It is stable in air but darkens in colour upon exposure to strong sunlight. HTC is stable in acid solutions having a pH higher than 2. It is capable of forming chelate complexes with metal ions.

Demeclocycline (DMC) and Doxycycline (DC) are yellow crystalline powders. It is odourless and has a bitter taste. It is sparingly soluble in water. It has no antibiotic spectrum similar to that of other tetracyclines. But it is slightly more reactive than the other against most of the micro organisms. It is officially used for the treatment of various types of bacterial infection. Its use as an antibiotic is particularly significantly in lime disease, acne and bronchitis, vanadium has been found to play a number of roles in biological systems. It is present in certain vanadium dependent haloperoxidases and nitrogenase enzymes. The mushroom amanita muscaria accumalate vanadium in the form of a co-ordination complex called amavadin whose function is still unknown one.

2. MATERIALS AND METHODS

Spectral measurements are performed on an Elico SL UV-visible spectrophotometer. The pH measurements were made using an Elico pH meter.

Double distilled water is employed for the preparation of solutions. All chemicals and reagents used for these studies are analytical grade obtained from Merck. The drugs are obtained from Sigma.

The standard solution of drugs were prepared in double distilled water.

3. RECOMMENDED PROCEDURE

Known aliquots of the buffer solution of required pH, vanadium(V) solution and drug solution were pipetted into 25ml standard flask. The contents of the flask is made up to the mark with double distilled water and shaken well for uniform concentration. The absorption spectra recorded against the respective blank solution.

4. RESULTS

4.1 Effect of pH

Absorption spectral characteristics were studied in the pH range 1 to 8 and the absorption spectrum of the complex recorded in the range 350 to 700nm. The DMC-V(V) complex exhibits maximum absorbance at 407.3nm at pH-5 (Fig-1) and those corresponding to DC-V(V) complex are 398.8nm at pH-4 (Fig-2) and HTC-V(V) complex are 401.0nm at pH-4 (Fig-3).

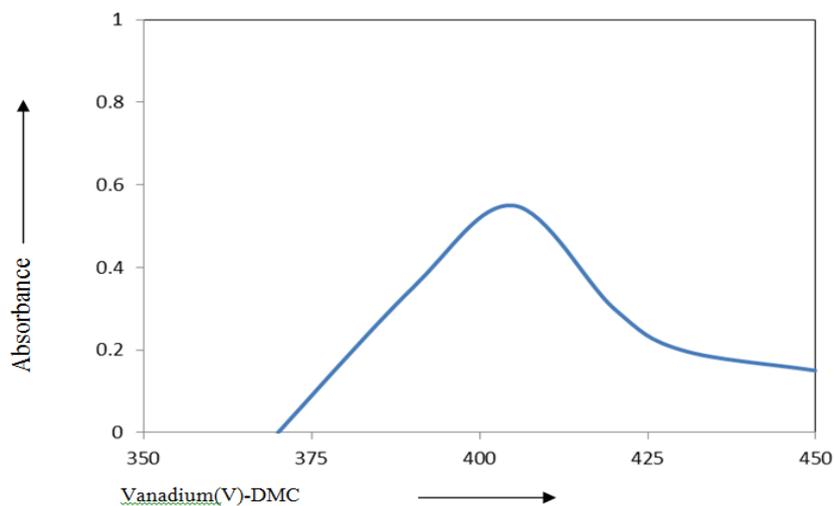


Fig-1: Absorption spectrum of V(V) - DMC system.
 $p^H = 4.0$; $[V(V)] = 4 \times 10^{-4} M$; $[DMC] = 0.06 \text{ mg/ml}$

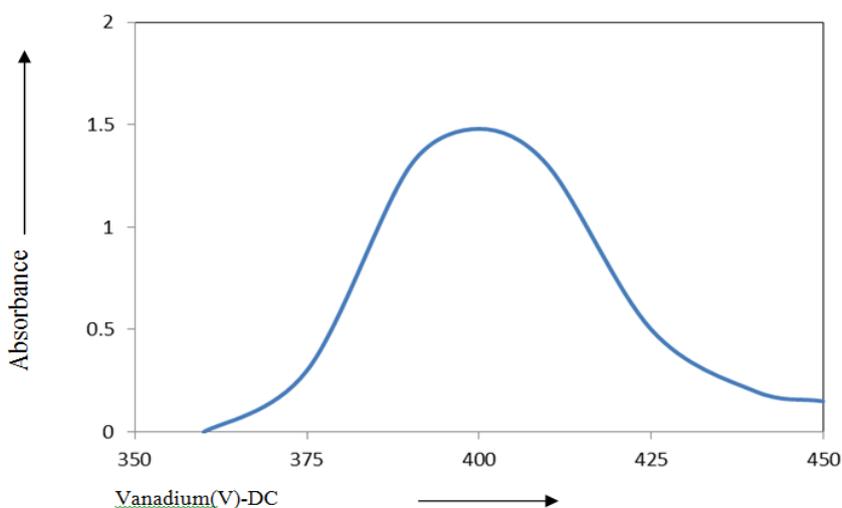


Fig-2: Absorption spectrum of V(V) - DC system.
 $p^H = 6.0$; $[V(V)] = 4 \times 10^{-4} M$; $[DC] = 0.06 \text{ mg/ml}$

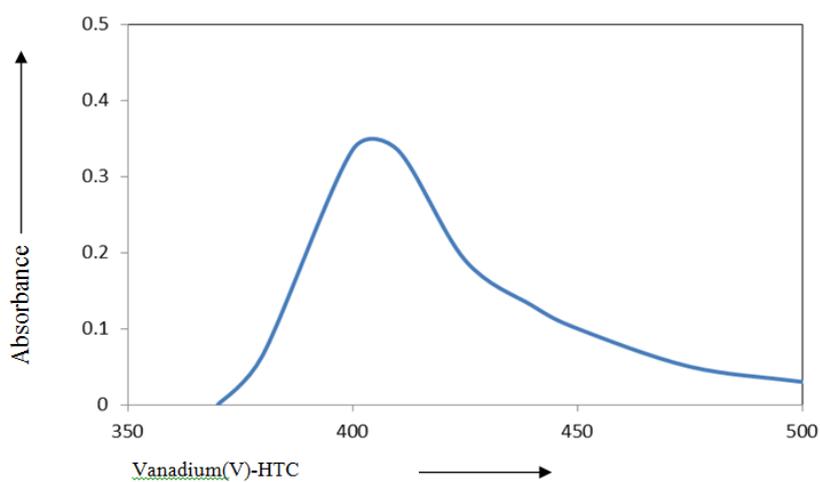


Fig-3: Absorption spectrum of V(V) - HTC system
 $p^H = 4.0$; $[V(V)] = 4 \times 10^{-4} M$; $[HTC] = 0.06 \text{ mg/ml}$

4.2 Effect of Time

The absorbance values of the complex solution are recorded over a period of two hours at regular intervals of time. The absorbance values are found to be approximately constant indicating that the complex formed is quiet stable over a period of 2 hrs.

4.3 Effect of Metal ion concentration

The concentration of the drug was maintained constant. Studies relating to the effect of metal ion concentration were carried out by varying the V(V) concentration. The linear calibration plots are shown in Fig-4, Fig-5 and Fig-6 respectively for DMC, DC and HTC.

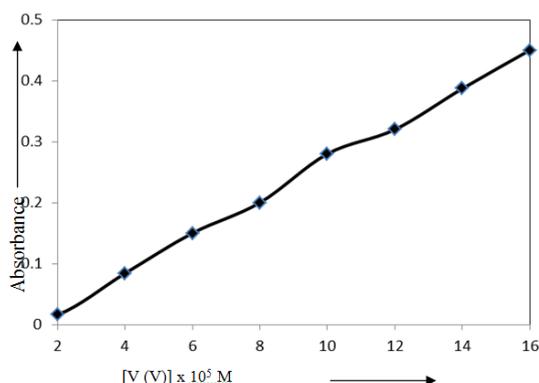


Fig-4: Effect of [V(V)] on absorbance
p^H = 4.0; [DMC] = 0.06 mg/ml; λ_{max} = 407nm

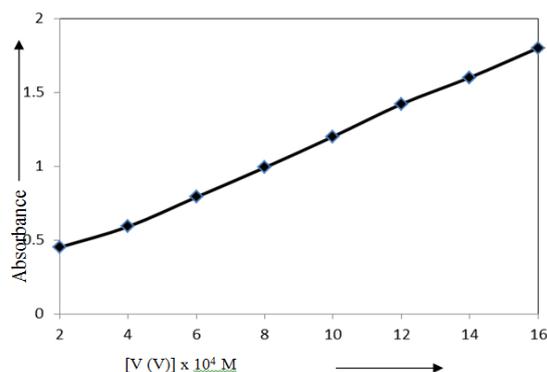


Fig-5: Effect of [V(V)] on absorbance
p^H = 5.0; [DC] = 0.06 mg/ml; λ_{max} = 399nm

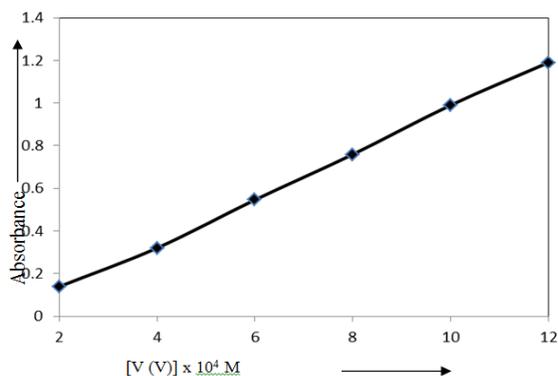


Fig-6: Effect of [V(V)] on absorbance
p^H = 4.0; [HTC] = 0.06 mg/ml; λ_{max} = 401nm

4.4 Analytical determination of Drug

Under the established optimum conditions a calibration plot was constructed by varying the concentration of the drug. The linear calibration plots shown in the Fig7, Fig-8 and Fig-9 indicate that the drug can be determined in the range of 0.01 to 0.06 μg/ml for DMC, 0.01 to 0.08 μg/ml for DC and 0.01 to 0.06 μg/ml for HTC.

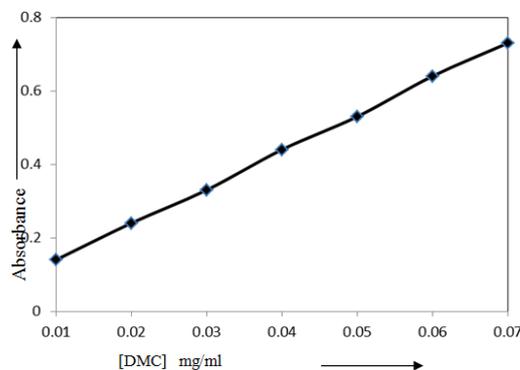


Fig-7: Effect of DMC on absorbance
p^H = 4.0; [V(V)] = 4x10⁻⁵ M; λ_{max} = 407 nm

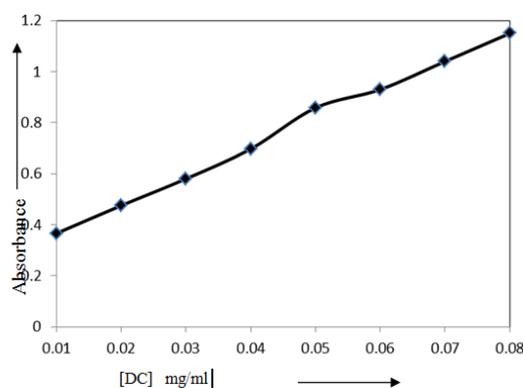


Fig-8: Effect of DMC on absorbance
p^H = 5.0; [V(V)] = 4x10⁻⁴ M; λ_{max} = 399 nm

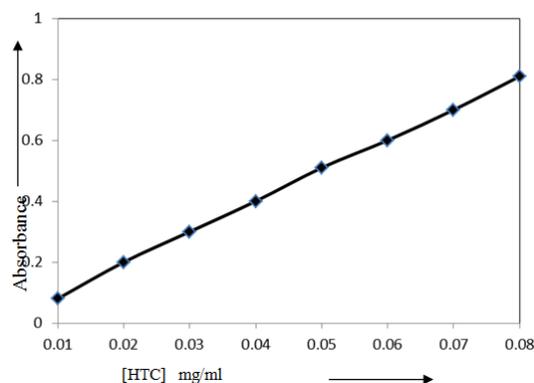


Fig-9: Effect of HTC on absorbance
p^H = 4.0; [V(V)] = 8x10⁻⁴ M; λ_{max} = 401 nm

5. COMPOSITION OF THE COMPLEX

The complex solution exhibits a colour in the case of DMC, DC and HTC. The author conducted Job's method

of continuous variation to determine the stoichiometric ratio of drug to vanadium(V). The corresponding Job's

curves are shown in Fig-10, Fig-11 and Fig-12 for DMC, DC and HTC respectively.

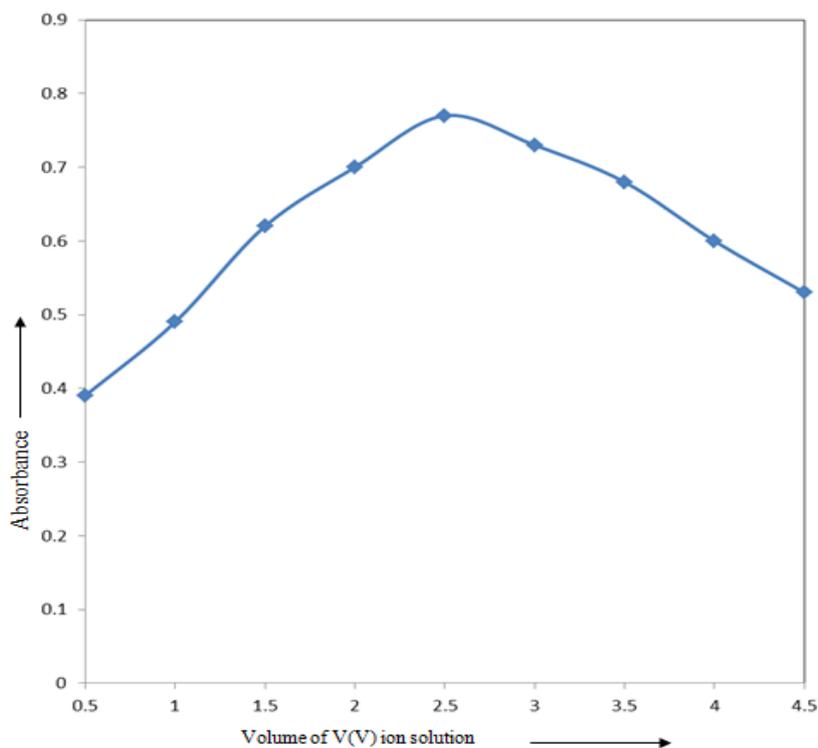


Fig-10: Job's continuous variation method
pH = 5.0; [V(V)]= 1×10^{-3} M; [DMC]= 1×10^{-3} M; $\lambda_{\max} = 407$ nm

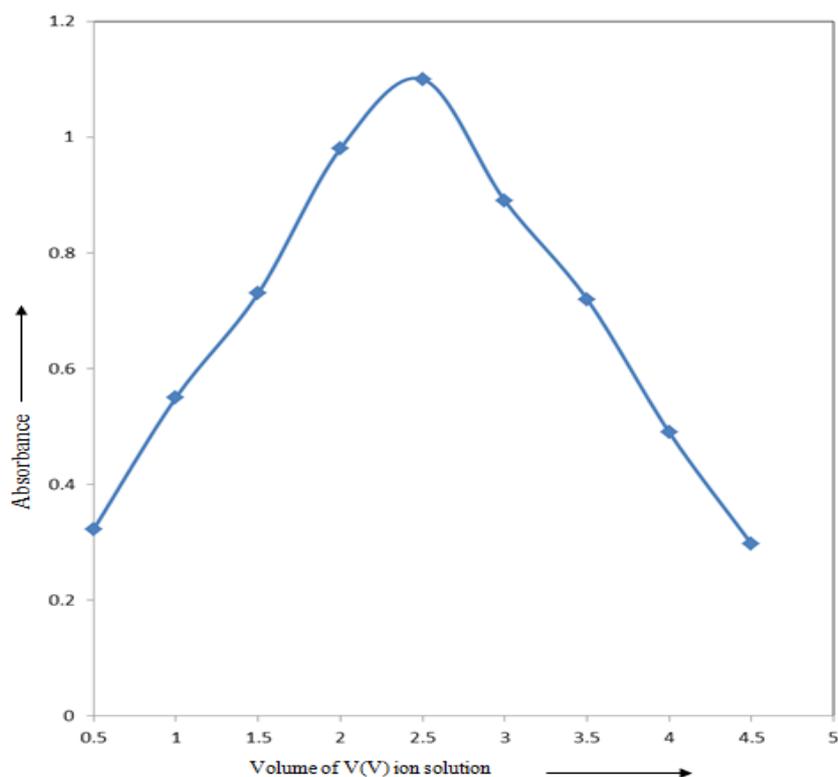


Fig-11: Job's continuous variation method
pH = 5.0; [V(V)]= 1×10^{-2} M; [DC]= 1×10^{-2} M; $\lambda_{\max} = 399$ nm

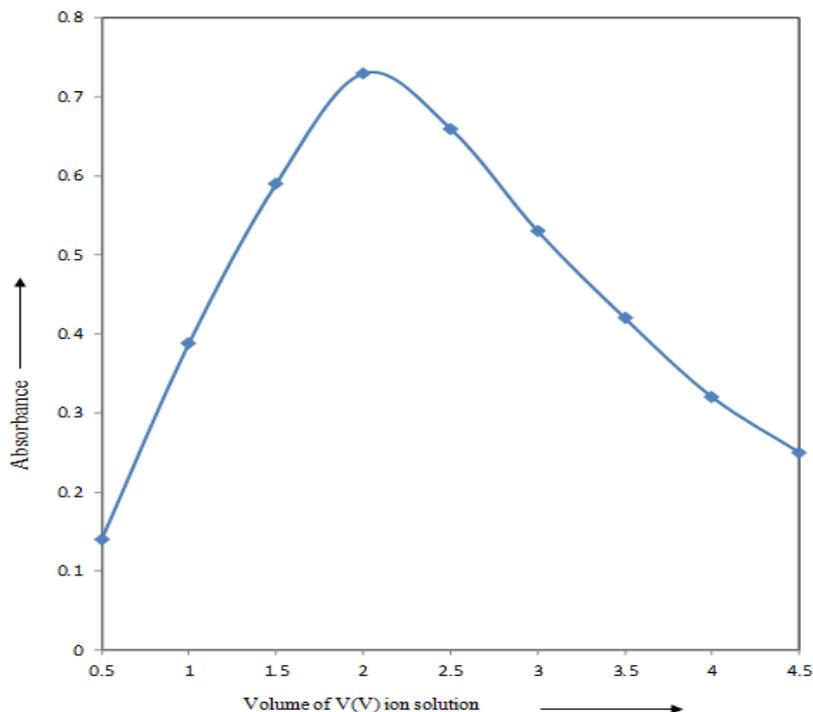


Fig-12: Job's continuous variation method
 pH = 4.0; [V(V)]= 1×10^{-2} M; [HTC]= 1×10^{-2} M; λ_{\max} = 401 nm

6. INTERFERENCE STUDY

The effect of interfering ion on the determination of V(V) was investigated that 10 fold excess of cobalt(II), uranium(VI), nickel(II) and iron(II) and iron(III) do not cause any interference in the determination of V(V). Similarly anion like Fluoride, Chloride, Sulphate, Oxalate show no interference to the extent of 50 fold vanadium(V).

7. CONCLUSION

The proposed method is simple and sensitive for the determination of vanadium(V) using DMC, DC and HTC in acidic medium.

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