



A spectrophotometric method for the determination of demeclocycline and zirconium (IV)

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ABSTRACT

Analytical determination of Demeclocycline (DMC) and Zirconium (IV) in pharmaceutical formulation is carried and based on a colour reaction between DMC and Zirconium (IV) forms yellow coloured complex with DMC in a buffer of P^H -6. The method can be conveniently used for the determination of Zirconium (IV) and DMC in the ranges of 0.95 to 7.35 mg/ml. and 0.004 to 0.040 mg/ml respectively. The method is successfully applied for the determination of DMC in pharmaceutical formulation. Effect of various parameters in his pH, metal ion concentration, drug concentration, of vary solvents is studied. The optimum condition are observed for the determination of zirconium and the drug.

Key words: Spectrophotometric method, DMC and Zirconium system.

INTRODUCTION

Demeclocycline (DMC) is a yellow crystalline powder. It is odourless and has a bitter taste. It is sparingly soluble in water. It has an antibiotic spectrum similar to that of other tetracycline's 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 infections. Its use as an antibiotic²⁻³ is particularly significantly in lime disease acne and bronchitis, Zirconium, strong ductile metal is having excellent resistance to corrosion. It is found to exist mostly in +4 oxidation state. It is observed that Zr^{+4} has a strong tendency to add on negative ions thus making its determination more difficult.

Preparation of the solution

The standard solutions of metal ions are prepared by exactly weighing the required amount of their salts in analytical balance, transferring it to a standard flask, dissolving and making up to mark using double distilled water.

The drug solution is prepared by dissolving exactly 50 mg of the demeclocycline in 100 ml volumetric flask. About 0.5 ml of con H_2SO_4 is added and the solution is made up to the mark with double distilled water.

Digital pH meter model CL21 of Elico company is used for the preparation of buffer solutions. Elico micro processor (μp) based double beam Uv-VIS spectrophotometer SL164 is used to measure absorbance of the solution chosen for study.

MATERIALS AND METHODS

1ml of 10^{-2} M Zirconium⁷ solution and 10 ml of buffer solution of required pH are taken in a 25 ml standard flask. To this 4 ml of freshly prepared demeclocycline solution is added and the contents are made up to the mark with

double distilled water. The spectra of the test solutions are recorded against the respective blank solutions containing two drug.

RESULTS AND DISCUSSION

4.1 EFFECT OF P^H : The effect of pH on the complexation reaction is studied. The absorbance values increase from pH 2 to 6 and then onwards there is a decrease. In solutions of high acidity as well as high alkalinity, the absorbance values are less. Keeping in view the reproducibility of the results and stability of solutions, pH 6 is chosen for further studies. The maximum absorbance is noticed at 420nm.

4.2 EFFECT OF METAL ION CONCENTRATION: The effect of Zirconium concentration on the absorbance is studied. The concentration of Zirconium is increased from 1 to 8×10^{-5} M. The concentration of demeclocycline⁴⁻⁶ is kept constant at 0.04 mg/ml. The P^H of the solution is maintained at 6. From the result it may be concluded that Zirconium⁷ can be determined in the range 0.91 to 7.35 $\mu\text{g/ml}$

4.3 EFFECT OF DRUG CONCENTRATION: The effect of demeclocycline concentration is studied in the range 0.004 to 0.040 mg/ml. The absorbance values are measured at 420 nm. The concentration of Zirconium is 8×10^{-5} M. Using the present method the drug can be determined in the range 0.004 to 0.040 mg/ml. It is shown in figure.1.

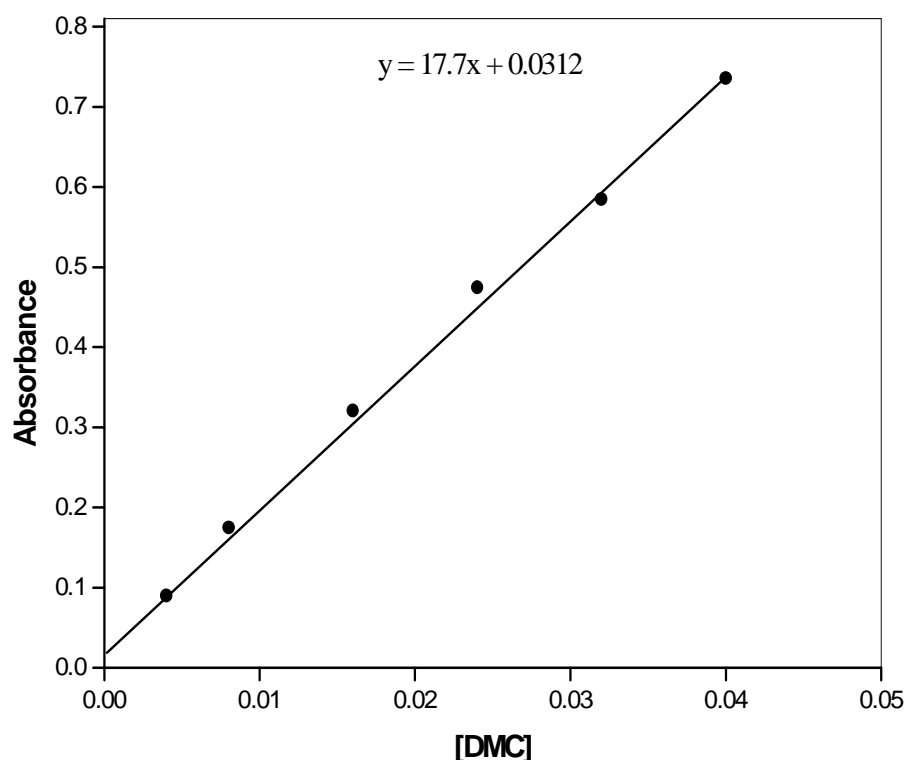


Fig. 1. Effect of [DMC] on absorbance
 $pH = 6$, $[Zr(IV)] = 8 \times 10^{-5} M$, $\lambda_{max} = 420nm$

4.4 EFFECT OF TIME ON THE REACTION: The effect of time on absorbance is studied for a period of 90 minutes for the same solution at regular interval of 15 minutes. It is found that the absorbance values remain constant for at least 45 minutes and then decrease to a small extent.

4.5. EFFECT OF ORGANIC SOLVENTS: Organic solvents generally influence a complexation reaction. Therefore effect of methanol, dimethylformamide, acetonitrile and dioxane (50% by volume) is investigated. An analysis of data reveals that there is an appreciable decrease in absorbance when organic solvents are employed.

4.6. COMPOSITION OF THE COMPLEX: The stoichiometry of the Zr (IV)-DMC complex was determined by job's⁸⁻¹⁰ method of continuous variation. In Job's method a series of solutions containing varying volumes of metal ion and DMC solution of equimolar concentration were taken in 10 ml volumetric flask. The absorbance values of these solutions were recorded in each case and the data is presented in fig. 2 It is clear from the figure that Zirconium forms 1:1 complex with the reagent.

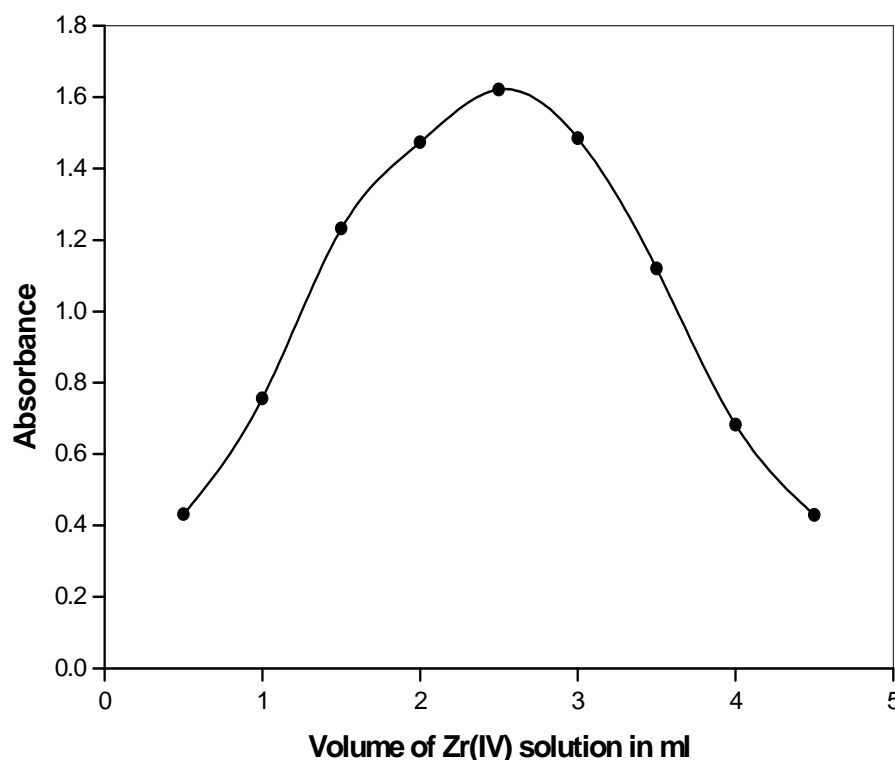


Fig. 2. Job's method of continuous variation
 $pH = 6$, $\lambda_{max} = 410nm$, $[Zr(IV)] = 1 \times 10^{-3}M$, $[DMC] = 1 \times 10^{-3}M$

5. EFFECT OF DIVERSE IONS:

The effect of interfering ion on the determination of Zirconium (IV) was investigated by adding known concentration of cobalt, manganese, chromium, cadmium, lead, cerium, a vanadium(IV) and uranium, Chromium and copper interfere when they are present to the extent of 70 fold, manganese to the extent of 100 fold and cobalt to the extent of 100 fold do not interfere in the determination. Metal ions like cerium, Iron, vanadium and tungsten seriously interfere, and cadmium and lead do not interfere. Anions such as fluorides, sulphate, acetate and oxalate interfere. When they are present to the extent of 70 fold Zirconium. The data is presented in table 1.

Table: 1 Interference Studies

Metal / Anion	Tolerance limit
Cd^{2+} , Pd^{2+} , and Co^{2+}	Do not interfere
Ce^{+4} , V^{+4} , V^{+5} , and W^{+6}	Seriously interfere
Cr^{+6} , and Cu^{2+}	Up to 30 fold
Mn^{+2}	Up to 100 fold
F^- , SO_4^{2-} , CH_3COO^- , $C_2O_4^{2-}$	Up to 70 fold

CONCLUSION

The proposed procedure is simple, sensitive and rapid. It is possible to determine the metal ion and demeclocycline in the range of 0.95 to 7.35 ($\mu g/ml$ and 0.004 to 0.040 mg/ml respectively).

Simple, selective and sensitive spectrophotometric methods is developed for the determination of demeclocycline. The procedure is based on the observation that DMC forms coloured complex with Zr(IV). The proposed methods can be employed for the analytical determination of metal ion, in the range of micrograms and DMC in milligram range. The method is successfully applied for the determination of DMC in pharmaceutical formulation.

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