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SPECTROPHOTOMETRIC DETERMINATION OF TETRACYCLINE IN SOME PHARMACEUTICAL PREPARATIONS

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ABSTRACT:

This paper briefly describes a simple spectrophotometric method for the determination of tetracycline in pharmaceutical preparation . The method is based on measurement of the absorbance of tetracycline- cerium(IV)complex at 430 nm in 1M sulphuric acid solution. Calibration graph for tetracycline was obtained over the range of 50-350 $\mu g.ml^{-1}$ The detection limit (2x noise) is 0.0025 $\mu g.ml^{-1}$ and with R.S.D for 5 replicate 0.03% .
This method allowed the determination of tetracycline in pharmaceutical preparations with a satisfactory accuracy when comparing it with official BP method.

Keywords; Spectrophotometry , cerium (IV) , Tetracycline , Pharmaceutical Preparation

INTRODUCTION :

Tetracycline Hydrochloride, (C_{22} H_{24} N_2O_8, HCL), yellow , odourless , hygroscopic , crystalline ,amphoteric powder with a bitter taste.Soluble in water free soluble in dilute acids insoluble in chloroform and ether and decompose in solutions of alkali hydroxides [1]. Tetracycline is a compound which belongs to a larg groups of antibiotics which contain four rings in their structures and capable of froming stable complexs with many metal ions [2]. Many methods have been reported for the determination of tetracyclines, including spectrophotometric [3], flow injection method [4]. Oxytetracycline hydrochloride(OTH) , like other tetracyclines , has been determined in variety of sample matrices [5, 6]. Many methods were used Ce(IV) forthe determination of drugs in pharmaceutical preparation and biological fluids [7-10].The present paper describes the using of Ce(IV) solution ,to develope spectrophotometric method for determination of tetracycline in pharmaceutical preparation .

EXPERIMENTAL:

Apparatus :All spectrophotometric measurements were made by Cintra 5 UV- Visible spectrophotometer.

Reagents and Chemicals

1- 1000$\mu g.ml^{-1}$ Ammonium cerium (IV) sulphate(Merck) solution was prepared by dissolving 1g of this material and dilute to 1000 ml with 1 M sulphuric acid .
2- 1 M sulphuricacid prepared by diluting 5.5 ml of concentrated- sulphuric acid to 100 ml by distilled water.
3- 1000 $\mu g.ml^{-1}$ tetracycline stock solution was prepared by dissolving 1 g of the tetracycline powder in 1000 ml. Standard solutions were prepared by an approirate dilution of stock solution .

Results and Discussion:

Tetracycline reacts with ammonium cerium (IV) sulphate to form a brown stable complex .It is suggested that Ce(IV) ion chelates with tetracycline. This can be used for the spectrophometric determination of tetracycline by measuring absorbance at 430 nm. The intensity of the colour formed depends very much on optimizing of the reaction conditions. A series of experiments were carried out to establish the optimum analytical variable for tetracycline determination such as the effect of Ce(IV) concentration, types of acid and acid concentration.

The Effict of Ce(IV) Concentration

The effect of Ce (IV) concentration at various sulphuric acid concentrations on 80µg.ml^{-1} of tetracycline is shown in Fig.1 . The results showed that the cerium(IV) and sulphuric acid as they were increasing the absorbance reading was increased to give a maximum absorption at 2.5 x 10^{-3} µg.ml^{-1} after which the signal started to decrease . The dilution efficiency is being the limiting factor . Therefor Ce(IV) concentration of 2.5 x 10^{-3} µg.ml^{-1} was chosen for further studies.

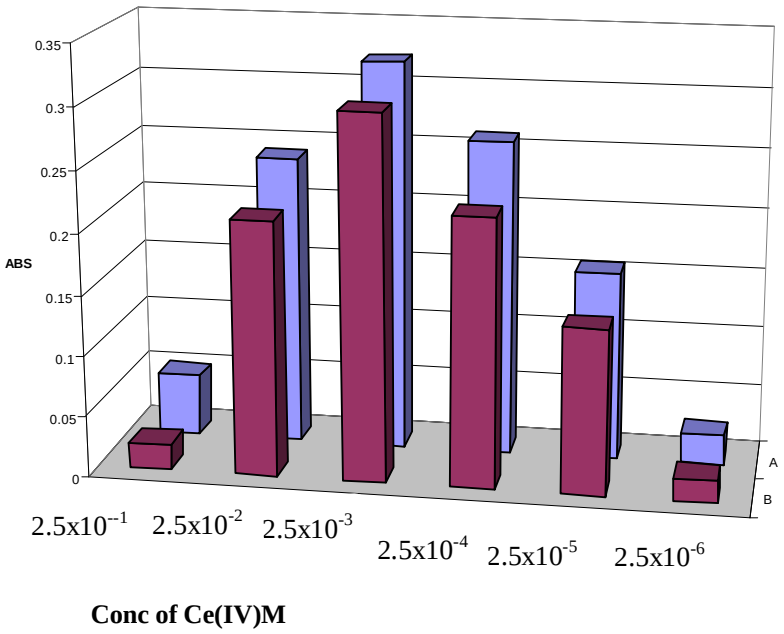

Fig . 1: Effect of Ce(IV)Concentration dissolved in tow different sulphuric acid concentrations ;(A) 1M and (B) 0.5M on the absorbance measurements of 80 µg.ml^{-1}tetracycline

Types of Acids

It was noticed that the sensitivity deteriorated when dilute hydrochloric acid was used instead of sulphuric acid due to forming turbid and unstable product. Therefore, sulphuric acid was found a suitable medium for sensitive measurements of tetracycline.

The Effect of Acid concentration

The effect of acid concentration on the absorbance was studied using different concentrations of sulphuric acid as shown in Fig.2. The low acidity of sulphuric acid less than 1 M hydrolyses the Ce(IV) and high acidity more than 1M protonates the tetracycline

molecule resulting in gradual decrease of the absorbance . Therefore, 1M sulphuric acid used for all further works.

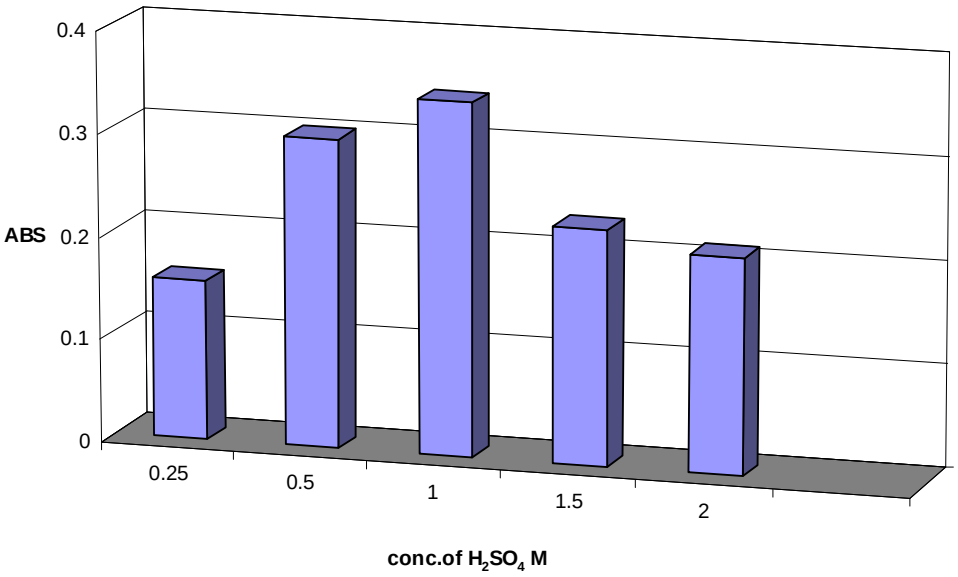

Fig. 2: Effect of H_2SO_4 concentration on the Absorbance measurements of 80 µg.ml^{-1} tetracycline in presence of 2.5x10^{-3} M Ce(IV) solution .

THE CALIBRATION DATA:

Calibration graph for tetracycline was obtained with the range 50-350 µg.ml^{-1} the equation for the best straight line where(y = 0.0004x- 0.0052) and the correlation coefficient is 0.988 . The detection limit(2xnoise) was 0.0025 µg.ml^{-1} and R.S.D is 0.03 % for 5 replicates determinations of 80µg.ml^{-1} tetracycline as shown in Fig.3.

APPLICATIONS:

The accuracy of this spectrophotometric method was tested by analysing three pharmaceutical dosage forms, capsules and solution containing tetracycline[11]. The results are summarized in Table 1, most of them agreed with reported values. Job method was used ,the molar formula is M_2L_3 as shown in Fig. 4.

In order to establish the validity of the proposed spectrophotometric method, the proprietary drugs containing tetracycline listed in Table 2 were analysed . The same batch of samples was analysed by BP method and the recoveries and R.S.D were calculated as shown in Table 2. The statistical analysis of these results reveals that there is no significant differences between them .This method offered a simple , accurate and direct method for tetracycline determination in pharmaceutical preparations.

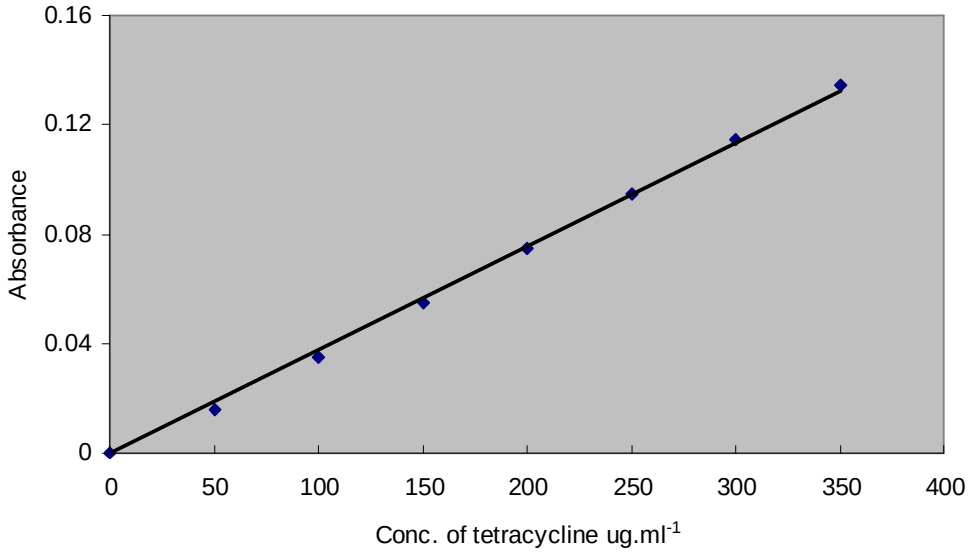

Fig. 3 : Calibration curve for determination of tetracycline by spectrophotometric method

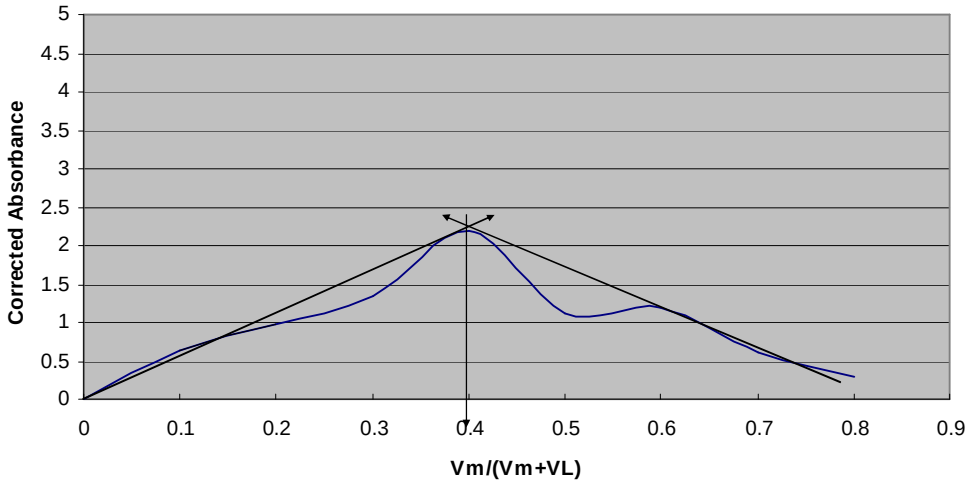

Fig.4:Method of continuous variation (Job method)

Table 1: Determination of Tetracycline in Drug Formulation by spectrophometric method .

Drugs (SDI)	Claimed $\mu g.ml^{-1}$	Found $\mu g.ml^{-1}$	Recovery%
OTOCAINE ear drops (100)mg	80.0	80.0	100.0
SAMACYCLINE capsules (250)mg	80.0	80.1	100.1
TETRACYCLINE capsules (250)mg	80.0	80.1	100.1

Table 2: Determination of Tetracycline in Drug Formulation by spectrophometric and B.P method .

Drugs (SDI)	BP methods %recovery±r.s.d% n=5	Spectrophotomatric method Recovery%± r.s.d% n=5
OTOCAINE ear drops (100)mg	99.0±0.01	100.0±0.04
SAMASYCLINE capsules (250)mg	101.0 ± 0.01	100.1 ± 0.03
TETRACYCLINE capsules (250)mg	103.0 ± 0.04	100.1 ± 0.04

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التقدير الطيفي للتتراسايكلين في بعض المستحضرات الصيدلانية

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البصرة–العراق

الخلاصة :

يصف البحث باختصار طريقة طيفية بسيطة لتقدير التتراسايكلين في بعض المستحضرات الصيدلانية.الطريقة تعتمد على امتصاصية معقد التتراسايكلين–السير يوم الرباعي عند 430 نانومتر في محلول1 مولارى من حامض الكبريتيك وتم الحصول على منحنى المعايرة للتتراسايكلين بحدود 350-50 مايكروغرام/مل مع حد كشف (2xالضوضاء) 0.0025 مايكروغرام/مل ومعدل الانحراف القياسي النسبي لخمس قرأءات 0.03%. هذه الطريقة تسمح بتقدير التتراسايكلين في المستحضرات الصيدلانية وبدقة الطريقة العالميةالبريطانية.