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Original Research Article

Determination of Chloramphenicol in Pharmaceutical Preparations and Environmental Wastewater Samples

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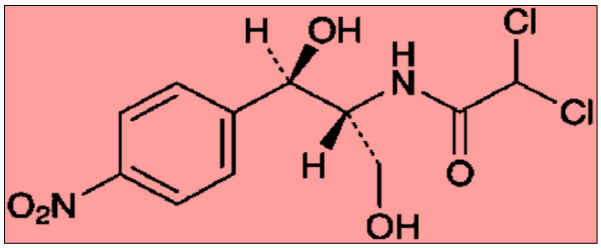
Abstract: A simple, precise, accurate, rapid, economical and sensitive Uv spectrophotometric method has been developed for the determination of Chloramphenicol in pharmaceutical preparations and environmental wastewater samples, which shows maximum absorbance at 281 nm in a solution of methanol**:** H_2O (10 in 1000) (v/v) was used as a solvent. Beer's law was obeyed in the range of 0.05 - 0.6 mg/ ml, with molar absorptivity of 6.6×10^5 $L.mol^{-1}.cm^{-1}$, relative standard deviation of the method was less than 1.6%, and accuracy (average recovery %) was 100 ± 1.0. The method was successfully applied to the determination of Chloramphenicol in some pharmaceutical formulations (Eye drops 0.5% and Eye Ointment1%) and industrial wastewater samples. The proposed method was validated by sensitivity and precision which proves suitability for the routine analysis of Chloramphenicol in true samples.

Keywords: Chloramphenicol, Pharmaceutical, Spectrophotometry, Environmental Samples.

INTRODUCTION

Chloramphenicol is chemically known as: is 2, 2-dichloro-N-[(1R,2R)-2-hydroxy-1-(hydroxymethyl)-2-(4- nitrophenyl) ethyl] acetamide, as shown in figure [1].



$C_{11}H_{12}Cl_2N_2O_5$**:** 323.13
2,2-Dichloro-N-[(1R,2R)-1,3-dihydroxy-1-(4-nitrophenyl) propan-2-yl] acetamide

Figure 1: Chemical structure of Chloramphenicol

Chloramphenicol a white, greyish-white or yellowish-white, fine, crystalline powder or fine crystals, needles or elongated plates, slightly soluble in water, freely soluble in alcohol and in propylene glycol. Chloramphenicol is an antibacterial which was first isolated from cultures of *Streptomyces venezuelae* in 1947 but is now produced synthetically. Chloramphenicol is an antibiotic with a bacteriostatic action which has a wide range of antimicrobial activity similar tetracycline hydrochloride but including also salmonella btyphi and S. Paratyphi and used in musculoskeletal and joint disorder such as rheumatoid arthritis, osteoarthritis and primary dysmenorrheal [1-6]. Different methods for the determination of Chloramphenicol, HPLC [7-11], gas chromatographic method [12]. LC-MS/MS methods [13-14]. Spectrophotometry methods [15-17]. Volta metric Method [18], chemiluminescences [19, 20]. However, all of these methods suffer from one or more disadvantage such as in sufficient sensitivity, selectivity, tedious and use of complex solvent extraction procedures. Therefore, a simple method for assay of Chloramphenicol is necessary for routine analysis and quality evaluation. It has the advantages of being rapid, sensitive, selective, accurate and reproducible. The present paper reports the development of a new UV method for determination of Chloramphenicol in different type of tablets, capsules and environmental water samples.

Experimental:

Apparatus

Shimadzu UV- 1700 pharma spec (double beam) spectrophotometer with 1.0 cm quartz cells was used for absorption measurement.

Reagents

All chemical used were of analytical or pharmaceutical grade and Chloramphenicol standard material was provided from AL-hokamaa company for pharmaceutical industries (HPI) Mosul-Iraq.

Methanol: H2O (10 in 1000) (v/v) was used as a solvent.

Chloramphenicol Standard Solution 25 Ppm

This solution was prepared by dissolving25 mg of Chloramphenicol in1000 ml of a solution of methanol: H_2O (10 in 1000) .in calibrated flask.

Determination of Absorption Maxima

The standard solution of Chloramphenicol (0.2 mg/ml) was scanned in the range of 220-350 nm which shows maxima located at 281 nm (Figure.1). Therefore, 281 nm wavelength was selected for the construction of calibration curve.

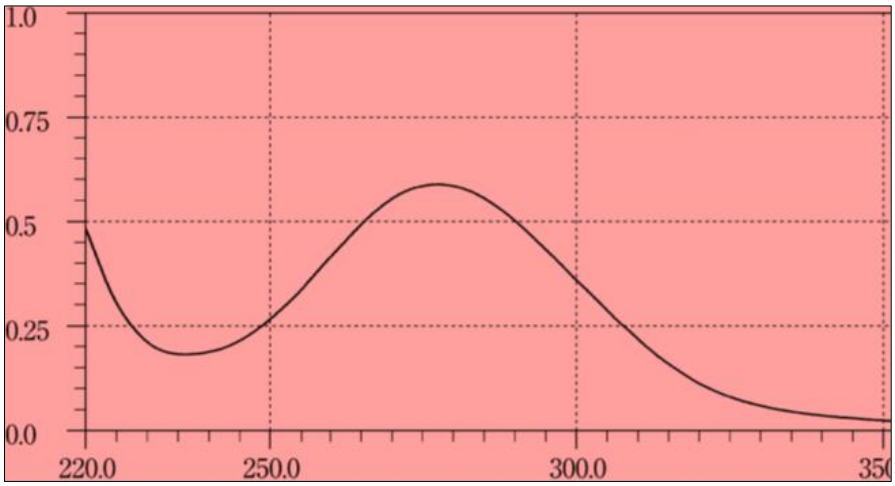


Figure 1: Absorption spectra of 0. 2mg/ml Chloramphenicol

Recommended Procedure

From the absorption maxima, calibration curve was prepared in the concentration range of 0.05-0.6 mg/ ml. The absorbance was measured at 281 nm against methanol: H_2O (10 in 1000) (v/v) was used as a solvent and blank. The concentration of the sample solution can be determined by using the calibration curve.

Analysis of Pharmaceutical Preparations

Procedures for Pharmaceutical Preparations (Eye Ointments)

Suspend a quantity of the ointment containing 1% gm. of Chloramphenicol in 50 ml of in 50 ml of petroleum spirit (boiling range, 40° to 60°) and extract with successive quantities of 50, 50, 50 and 30 ml of warm water. Combine the extracts, dilute to 200 ml with water, mix well and filter, discarding the first 20 ml of the filtrate. Dilute 10 ml of the filtrate to 50 ml with water and measure the absorbance of the resulting solution at the maximum at. 281nm using distilled water as blank and the concentration was calculated by using the calibration curve of this method.

Procedures for Pharmaceutical Preparations (Eye Drops):

Dilute a quantity containing 5 mg of Chloramphenicol to 25 ml with water. Dilute 10 ml to 100 ml with water and measure the absorbance of the resulting solution at the maximum at 281 nm, using distilled water as blank and the concentration was calculated by using the calibration curve of this method.

Procedure for Real Water Samples

To demonstrate the practical applicability of the proposed method, real water samples were analyzed by this method. Industrial waste water from AL-hokamaa company for pharmaceutical industries (HPI) Mosul-Iraq, were fortified with the concentrations in the range of 1, 2, 4, 5, mg/ml of Chloramphenicol. The fortified water samples were analyzed as described above for recommended procedure and the concentration was calculated by using the calibration curve of this method.

RESULT AND DISCUSSION

UV- Visible spectrophotometry is still considered to be a convenient and low-cost method for the estimation of pharmaceuticals [21, 22]. This method used for the determination of Chloramphenicol in pharmaceutical preparations and environmental wastewater samples was found to be sensitive, simple, accurate, and reproducible. Beer s law was obeyed in the concentration range of 0.05-0.6 µg/ ml. (Figure 2) with correlation coefficient of 0.9985, intercept of 0.002 and slope of 2. 8427. The conditional molar absorptivity was found to be 6.6×10^5 x10^4 l/mol.cm

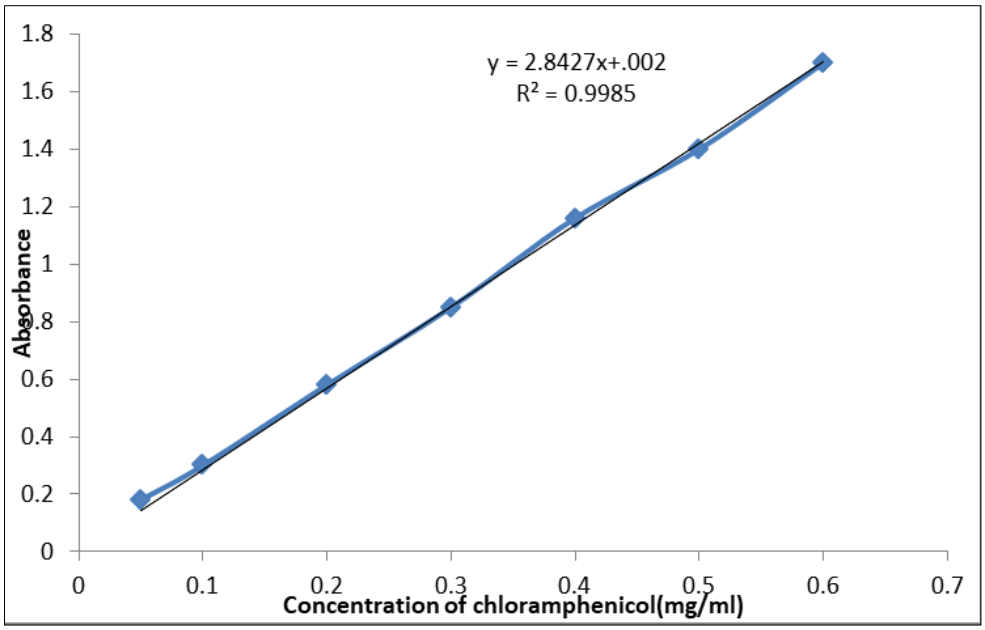


Figure 2: Calibration curve for Chloramphenicol

The accuracy and precision of the method, a pure drug solution was analyzed at three different concentrations, each determination being repeated six times. The relative error (%) and relative standard deviation values are summarized in (Table 1). From (table 1) the values of standard deviation were satisfactory and the recovery studies were close to 100%. The RSD% value is less than1.6 indicative of accuracy of the method.

Table I: Accuracy and precision of the proposed method.

Chloramphenicol taken (mg/ml)(	Er (%)[a]	RSD (%)
0.1	1.1	1.6
04..	1.2	1.6
0.6	1.2	1.5

a: Mean of six determinations.

The limit of detection (LOD) and limit of quantitation (LOQ) were calculated using the standard deviation of the intercepts (σ) and the mean slope(s) of the calibration curves. LOD=3.3σ/s and it was $0.7x10^{-3}$ mg/ml. and LOQ=10 σ/s and it was $2.1x10^{-3}$ mg/ml [23]. The results are compiled in [Table 2].

Table 2: Optical characteristics and statistical data for regression equation of the proposed method

Parameters	Value
λ max (nm)	281
Beer´s law limit (mg .ml^{-1})	0.05 - 0.6
Molar absorptivity (l.mol^{-1}.cm^{-1})	6.6×10^{5}
Correlation coefficient (r^2)	0.9985
Regression equation(Y= a × + b)	
Slope (a)	- 0.002
Intercept (b)	2.8427
Recovery %	100±0.4
Relative standard deviation (%)	< ±1.6
Limit of detection, (mg\ml)	$0.7x10^{-3}$mg/ml
Limit of quantification, (mg\ml)	$2.1x10^{-3}$ mg/ml

Application of the Proposed Method:

The proposed method was satisfactorily applied to the determination of Chloramphenicol in its pharmaceutical preparations [Ointments, Eye drops], and wastewater samples, the results of the assay of the pharmaceutical preparations revels that there is close agreement between the results obtained by the proposed method and the label claim [Table 3]. And the results of water samples [Table 4] show that the recovery values obtained were close to 100%.

Table 3: Assay of Chloramphenicol in pharmaceutical formulations

Pharmaceutical formulations	Amount of Chloramphenicol*	Label amount
Chloramphenicol Eye drops(0.5%) (HPI)	0.4998%	0.5%
Samaphenicol Eye (Ointments) 1% (NDI)	0.994%	1.0%

*Mean of five determinations.

Table 4: Determination of Chloramphenicol in industrial wastewater samples

Wastewater samples	Added mg/ml	Found* mg/ml	Recovery %(n=10)
Industrial wastewater	1	1.001	100.1
	2	2. 01	100.5
	4	4.02	100.5
	5	5.01	100.2

*Mean value of ten determinations.

CONCLUSION

The UV spectrophotometric method proposed is simple, sensitive, rapid, low-cost, does not involve solvent extraction steps and gives precise and accurate results. The proposed method was successfully applied to analysis of Chloramphenicol in [Eye Ointments, Drops] and environmental wastewater samples.

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