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Research Article

Spectrophotometric Estimation of Vardenafil Hydrochloride in Pharmaceutical Preparations and Environmental Wastewater Samples: Application to Content Uniformity Testing

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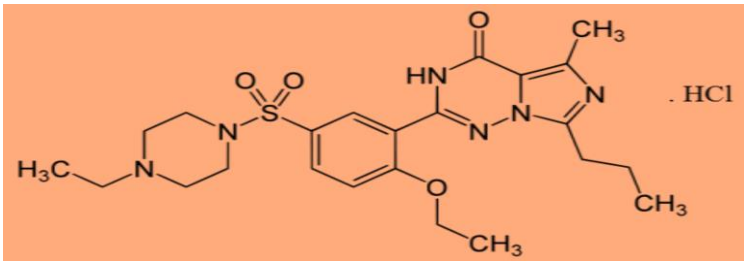
Abstract

Very Simple, accurate, precise, rapid, economical and high sensitive UV spectrophotometric method has been developed for the estimation of vardenafil hydrochloride in pharmaceutical preparations and environmental wastewater samples, which shows maximum absorbance at 244 nm in distilled water. Beer's law was obeyed in the range of) 0.1-2µg/ ml (with molar absorptivity of 4.745×105 L.mol-1.cm-1, relative standard deviation of the method was less than 1.6%, and accuracy (average recovery %) was 100 ± 1.0. No interference was observed from common excipients and additives often accompany with vardenafil hydrochloride in pharmaceutical preparations. The method was successfully applied to the estimation of vardenafil hydrochloride in some pharmaceutical formulations (tablets) and industrial wastewater samples. The proposed method was validated by sensitivity and precision which proves suitability for the routine analysis of vardenafil hydrochloride in true samples. Application to content uniformity testing

Key Words: vardenafil hydrochloride; spectrophotometry; pharmaceutical preparations; environmen samples

Introduction

Vardenafil hydrochloride (VAR) is chemically (1-[[3-(1,4-dihydro-5-methyl-4-oxo-7-propyli-midazo [5,1-f][1,2,4]triazin-2-yl)-4-ethoxyphenyl]sulfonyl]-4-ethylpipera-zine,mono hydrochloride (Figure.1) .Vardenafil hydrochloride is not official in any Pharmacopoeia , used to treat erectile dysfunction. Vardenafil inhibit phosphodiesterase type 5 (PDE-5) enzyme, which in turn maintains higher levels of cyclic guanosine monophosphate. Which relaxes smooth muscles, promotes penile blood flow and enhances erectile function[1-3]



C23H32N6O4S, HCl= 525.1

Figure 1: Chemical Structure of Vardenafil hydrochloride

Several methods for the determination of Vardenafil hydrochloride have been described in the literature, including spectrophotometric methods [4-9], capillary electrophoretic methods [10], stability indicating LC method [11], High performance liquid chromatographic methods [12-16], and atomic

emission and atomic absorption spectrometry method [17]. The present work describes a new, simple UV-spectrophotometric method for the determination of Vardenafil hydrochloride in pure form, pharmaceutical formulations and in industrial wastewater samples.

Experimental Methods

Apparatus

Spectro-scan 50 UV- visible (double beam) spectrophotometer with 1.0 cm quartz cells was used for absorption measurements

Reagents

All chemical used were of analytical or pharmaceutical grade and Vardenafil hydrochloride standard material was provided from (Al-Hokamaa Company for Pharmaceutical Industries (HPI) Mosul-Iraq), company.

Vardenafil hydrochloride standard solution.

This solution was prepared by dissolving 10 mg of vardenafil hydrochloride in 1000 ml of distilled water in calibrated flask.

Estimation of absorption maxima

The standard solution of vardenafil hydrochloride (1µg/ml) was scanned in the range of 200-350nm which show maxima located at 244 (Figure 2). The higher absorption value at 244 nm results in increasing sensitivity of the method at this maximum. Therefore, 244 nm wavelength was selected for the construction of calibration curve.

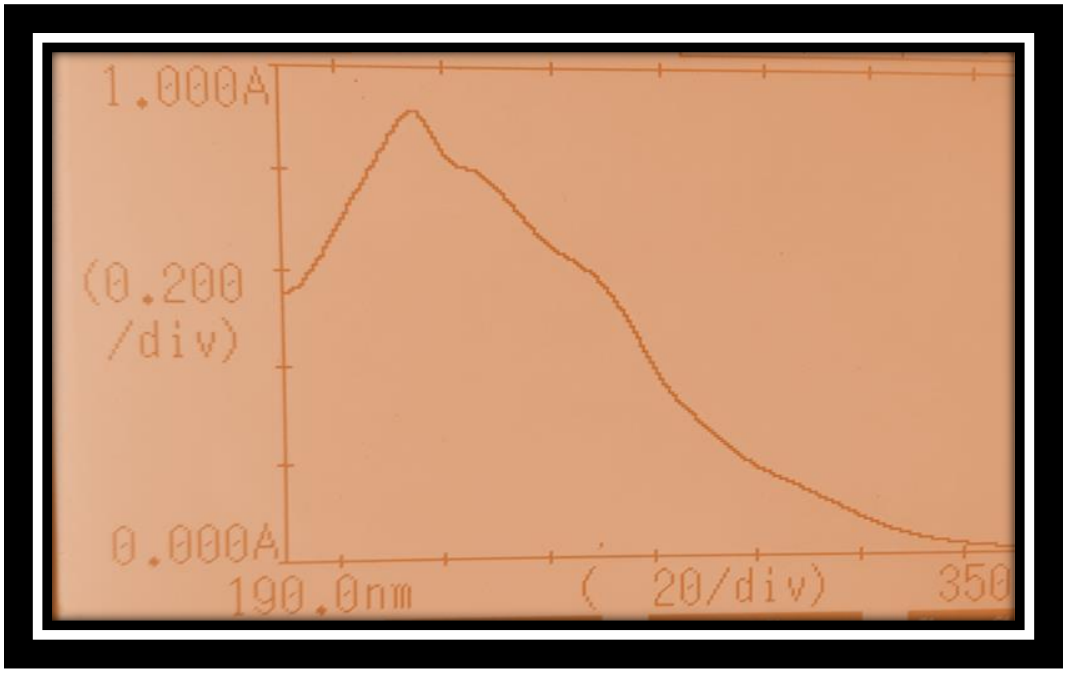


Figure 2: Absorption spectra of 1µg/ml vardenafil hydrochloride against distilled water.

Recommended procedure

From the absorption maxima, calibration curve was prepared in the concentration range of (0.1-2)µg/ml.The absorbance was measured at 244 nm against distilled water as a blank .The concentration of the sample solution can be determined by using the calibration curve .

Procedures for pharmaceutical preparations

For the estimation of vardenafil hydrochloride in tablet preparations ,and to minimize a possible variation in the composition of the tablets, the mixed content of 20 tablets of the brand , were weighed and grounded to fine powder, then the powder equivalent to 1 mg of vardenafil hydrochloride was stirred well with about 90 ml of distilled water for 20 minutes and the volume was made to 100mL with distilled water ,filtered through whatman No. 41 filter paper and 10 ml of this solution was diluted to 100 ml by distilled water to get 1µg/ml solution and aliquot of this solution was treated as described above for recommended procedure 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 the state company for drug industries and medical appliances Mosul-Iraq, were fortified with the concentrations in the range of (0.2,0.8,1.6) µg/ml of vardenafil hydrochloride .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. [18-22] This method used for the estimation of vardenafil hydrochloride in pharmaceutical preparations and environmental wastewater samples was found to be high sensitive, simple, accurate, and reproducible. Beer s law was obeyed in the concentration range of 0.1-2 µg/ml (Figure 3)with correlation coefficient of 0.9996,intercept of 0.0003 and slope of 0.9037. The conditional molar absorptivity was found to be (4.745x105) L.mol-1.cm-1 and sandell's sensitivity was(1.107ng.cm-2). The limit of detection and quantification were evaluated as: LOD =3.3xIntercept/10 And LOQ = 3LOD

Slope

The limit of detection was(0.109ng/ml) and the limit of quantification (0.327ng/ml) as the lowest standard concentration which could be determine with acceptable accuracy

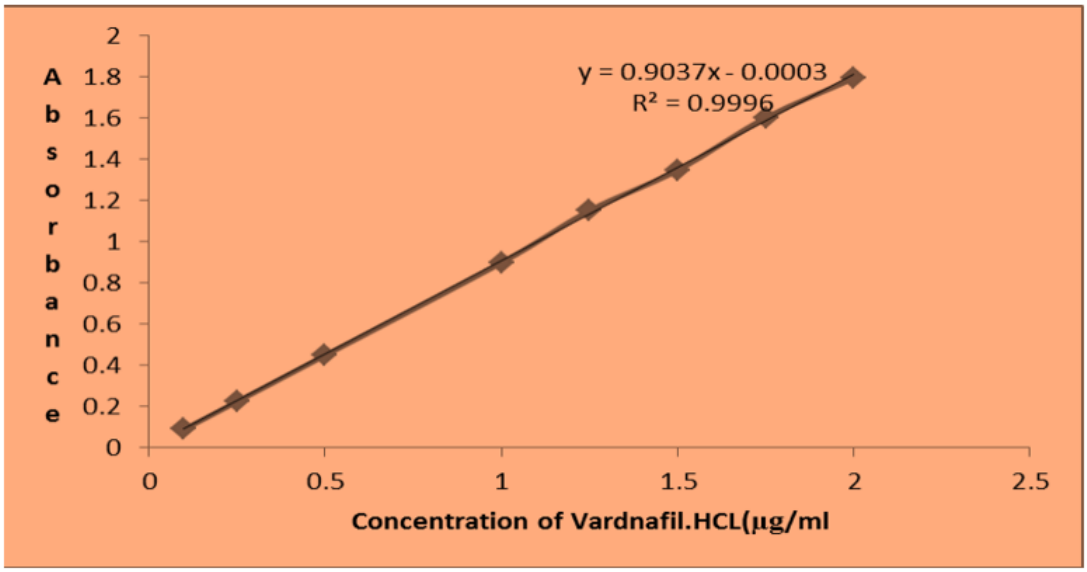


Figure 2: Calibration graph of vardenafil hydrochloride.

The accuracy and precision of the method, a pure drug solution was analyzed at three different concentrations, each estimation 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.

Vardenafil hydrochloride (taken µg/ml)	Er (%) [a]	RSD (%)
0.2	1.01	1.3
0.8	1.02	1.6
1.6	1.02	1.4

Table 1: Accuracy and precision of the proposed method. (a: Mean of six estimations)

Interference studies

In order to assess the possible applications of the proposed method, the effect of substance that often accompany with vardenafil hydrochloride in (Tablets) were studied by adding different amount of substances to 1 µg of vardenafil hydrochloride An attractive feather of the method is its relative freedom from interference by the usual diluents and excipients in amounts for in excess of their normal occurrence in pharmaceutical preparations. The results are given in (Table 2).

Interfering substances	Amount added/mg of interfering	Amount of drug found*µg	RSD %
Lactose	40	1.06	0.61
Microcrystalline cellulose	20	1.06	0.64
Corn starch	30	1.07	0.77
Povidone	30	1.05	0.78
Magnesium stearate	40	1.07	0.94
Hydroxyl propyl methyl cellulose	40	1.07	0.96
Poly ethylene glycol	20	1.01	0.95
Titanium dioxide	10	1.05	0.89

*Average of six estimations

Table 2: Estimation of 1 µg of vardenafil hydrochloride in the presence of excipients and other substances.

Analytical application

The proposed method was satisfactorily applied to the estimation of vardenafil hydrochloride in its pharmaceutical preparations tablets 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 closed to 100%.

Pharmaceutical formulations supplied by (HPI)Mosul-Iraq. Tablets	Label amount (mg/tab)	Found by proposed method (mg) *	Recovery%
Horse man tablet 20mg	20mg/tab	19.96	99.8

*mean value of ten estimations

Table 3: Estimation of vardenafil hydrochloride in pharmaceutical formulation

Wastewater samples	Added µg/ml	Found* (µg/ml)	Recovery %(n=10)
Industrial wastewater	0.2	0.202	101
	0.8	0.802	100.25
	1.6	1.606	100.375

* mean value of ten estimations.

Application of the method to content uniformity [23-27]

The proposed method proved to be suitable for the content uniformity test, where a great number of assays on individual tablets are required. Data presented in (Table5), indicate that the proposed method can accurately and precisely quantitate vardenafil hydrochloride in its commercially available tablets. The mean percentage (with RSD) of the labeled claim found in ten tablets was (0.14%) which fall within the content uniformity limits specified by the USP 33 [23].

Table [4]: Content uniformity testing of vardenafil hydrochloride tablets using the proposed method	% of the label claim
Tablet NO. 1	100. 16
Tablet NO. 2	100. 23
Tablet NO. 3	99. 88
Tablet NO. 4	100.41
Tablet NO. 5	99.38
Tablet NO. 6	99. 53
Tablet NO. 7	99.82
Tablet NO. 8	100. 15
Tablet NO. 9	100.26
Tablet NO. 10	100.16
Mean (x)	99.998
% RSD	0.41
Max. allowed unit [23]	±1.4%

Conclusion

The developed method is found to be high sensitive, accurate, simple, precise and economical, and can be used for routine quality control analysis of Vardenafil Hydrochloride in pure form, bulk, pharmaceutical formulations and environmental wastewater samples.

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