How to Cite:
Sadiq, W. J., & Hanoon, I. T. (2022). Using the zero-order derivative spectrum for the quantitative estimation of xylometazoline hydrochloride in its pure form and some of its pharmaceutical preparations. *International Journal of Health Sciences*, *6*(S4), 4168–4174. https://doi.org/10.53730/ijhs.v6nS4.9069

Using the zero-order derivative spectrum for the quantitative estimation of xylometazoline hydrochloride in its pure form and some of its pharmaceutical preparations

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***Abstract*---**A new spectrophotometric method has been developed for the rapid, accurate and inexpensive determination of xylometazoline (XMH) in its pure form and in some pharmaceutical preparations, using the first derivative technique. It was found that this technique enables us to quantitatively estimate xylometazoline in a range of concentrations (5-50 $\mu g.ml^{-1}$), the height of the peak above the base line of the wavelengths (268 and 292 nm) was taken advantage of. The obtained results showed that the method is accurate and has good agreement, as the percentage recovery was (95-100%), while the RSD value was (0.030-0.300%) for wavelengths respectively, the method was applied to a number of commercial pharmaceuticals successfully

***Keywords*---**xylometazoline, XMH, first derivative spectrum.

Introduction

Xylometazoline is a topical nasal decongestant that is fast-acting and highly effective in reducing nasal congestion. It is from the group of Imidazoline that works to remove the decongestant effect through adrenergic receptors that lead to constriction of blood vessels and thus resume the flow of nasal air [1,2] It is also used in the treatment of otitis media, which is often difficult to diagnose; There are no standard criteria or specific laboratory tests to diagnose it [3,4]. The drug is scientifically called 2- [(4-tert. butyl-2,6-dimethyl phenyl) methyl]-4,5-dihydro-1H-imidazole hydrochloride. Xylometazoline dissolves easily in water, ethanol, and methanol, and is used to treat stuffy or congested nose and minor swelling

caused by allergies or the common cold [5]. Figure 1 shows the chemical structure of the drug.

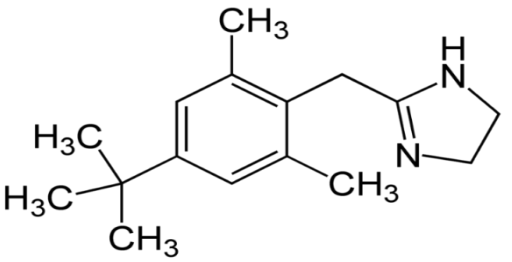


Figure 1: Chemical Structure of Xylometazoline

By reviewing scientific sources and references that are concerned with estimating medicines, we note the interest of a large number of researchers in quantitatively estimating Xylometazoline in its pure form and pharmaceutical preparations because of the medical importance of this drug, the drug was estimated by high-performance liquid chromatography [6-10], gas chromatography [11], and HPTLC [12], spectrophotometry was used to estimate the drug [13,14], and by using cyclic voltammetry, the drug was analyzed [15]. In this study, the derivative of the spectrum was used for quantitative estimation of Xylometazoline in some medical preparations. The method was accurate, sensitive, inexpensive, and did not require chemicals or pre-separation methods.

The practical part

Devices

In order to make the measurements, a dual-beam UV-Vis spectrophotometer, a Shimadzu-1650 type, a Canon 2900 laser printer, and a Germany-Sartorius sensor scale were used. The absorption spectra of the drug were recorded within the wavelength 190-350 nm, at a medium scanning speed, using a 1 cm quartz cell.

Solutions

To prepare 1000 $\mu g.ml^{-1}$, 0.05 gm of Xylometazoline (of Indian origin, obtained from the local market) is dissolved in an amount of distilled water that is added and completed to the mark in a volumetric 50 mL flask using the same solvent. A solution of 100 $\mu g.ml^{-1}$ of the pharmaceutical preparation (Otrivin/Drops 1mg GlaxoSmithKline, Egypt and DECOZAL/ Drops 1mg AMMAN PHARMACEUTICAL INDUSTRIES Jordan) was prepared by dissolving 1 ml of the pharmaceutical preparation in an amount of water and completing the volume in a 10 ml volumetric flask and completed to the mark using the same solvent.

The method of work

Various volumes of Xylometazoline hydrochloride standard solution (1-800 mg) were transferred into a series of 10 mL volumetric flasks and filled with distilled water to the mark. The absorption spectra were recorded against the mock

solution (distilled water) and then the required derivatization process was performed on the zero-order spectra to obtain the first derivative (5-50 μg.ml^{-1}).

Results and Discussion

Absorption Spectra

The absorption spectrum of Xylometazoline hydrochloride (5 μg.ml^{-1}) was recorded against the mock solution for a range of wavelengths ranging between 190-380 nm, at a medium scanning speed, with a rate of change was 0.1 nm and a bandwidth of 2 nm. Figure 2 shows the absorption spectrum of the drug, which shows two peaks at wavelength 197 and 257.9 nm.

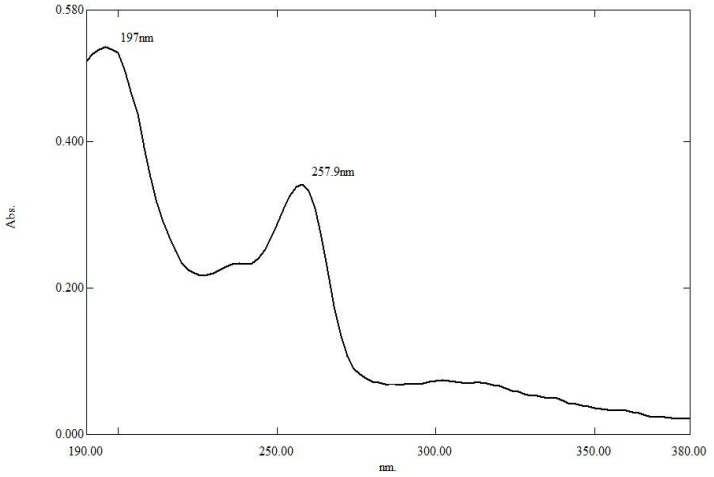


Figure 2: Xylometazoline absorption spectrum at a concentration (5 μg.ml^{-1}).

First Derivative Method

The spectral derivative technique is useful in determining the concentrations of substances required to be measured in the single estimation in the presence of drug additives and multiple mixtures, despite the presence of a large overlap between the absorption spectra [16], the first derivative was applied, and the results showed that the method can be applied successfully when using optimal conditions such as speed Scanning, rate of change and bandwidth. Multiple measurements were used on the spectra of the first derivative recorded under the optimum conditions for the spectrometer operation, which is the measurement of the height of the peak relative to the base line, for the purpose of quantitative analysis of the drug under study in its pure form and some of its pharmaceutical preparations. To prepare the first derivative, a spectrum of solutions of different concentrations (5-50 μg.ml^{-1}) of Xylometazoline hydrochloride was used.

The results of the measurements made based on these spectra showed that when changing the concentration of xylometazoline hydrochloride is directly proportional to the value of the measured peak height at wavelengths (268 and 292 nm). So, the spectra of the first derivative of Xylometazoline hydrochloride

were recorded, Figure 3 shows the spectrum of the first derivative for a series of different concentration.

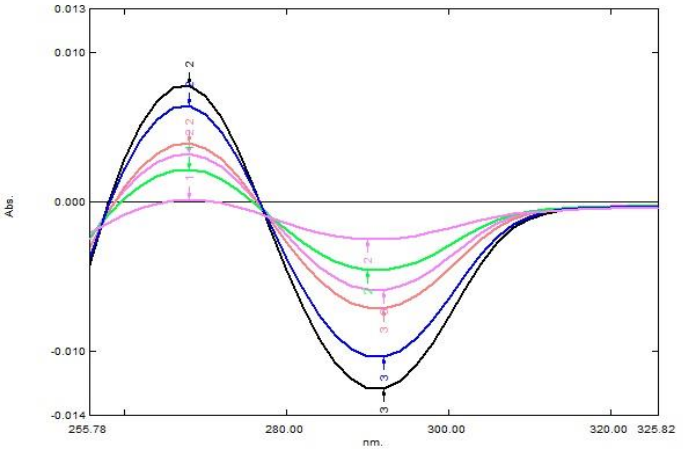
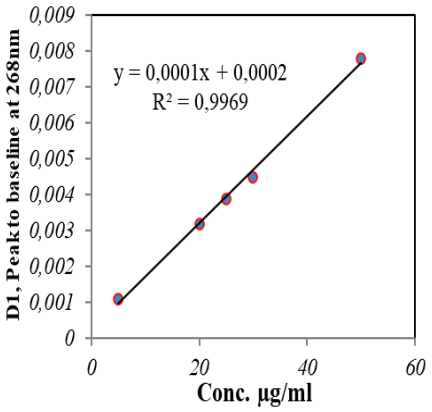


Figure 3: Spectrum of the first derivative of Xylometazoline at different concentrations.

Calculations and calibration curves

Considering the analytical characteristics and most of the statistical data for each of the proposed methods under optimal conditions, the linearity of the graphs of the calibration curves that ranged between (5-50 µg.ml^{-1}) was obtained, while the values of the estimating factor ranged (0.9969 and 0.9955) and the values of detection limits (4.67- 4.39) For the measurement areas indicated via a peak height above the base line (268 and 292 nm), respectively, Table 1 shows the equation of the straight line as well as the slope of the indicated areas.



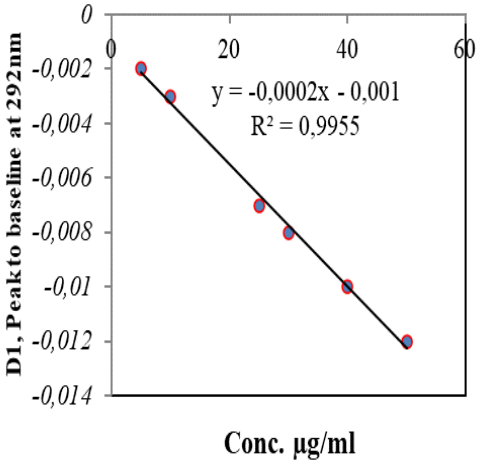


Table 1: Results of Xylometazoline assay by the first derivative

Compound	Order of derivative	Mode of calculation	Concentration range	λ (nm)	Regression question	R^2	LOD
Xylometazoline HCl	First	Peak to baseline	5-50 µg.ml^{-1}	268	y = 0.0001x + 0.0002	0.9969	4.67
				292	y = -0.0002x - 0.001	0.9955	4.39

Accuracy and Precision

The concentrations (10 and 20 µg.ml^{-1}) were chosen to find the accuracy and compatibility of the method, as the results showed that the method has good accuracy and precision, as the RSD% value ranged between (0.030-0.300%) and Rec% value was (95-100%) for Xylometazoline Hydrochloride, as shown in the table 2.

Table 2: Accuracy and Precision of the method

Compound	Order of derivative	Mode of calculation	Drug Conc. µg.ml^{-1}		Rec%	RSD%
			Taken	Found		
Xylometazoline HCl	First	Peak to baseline of 268nm	10	10	100	0.300
			20	20	100	0.250
		Peak to baseline of 292nm	10	10	100	0.100
			20	19	95	0.030

Method Application

The first derivative pattern was applied to the selected areas (peak height relative to base line) for direct determination of xylometazoline and it was successful. Table 3 shows the results of analyzing a number of different pharmaceutical preparations of the drug using the proposed method.

Table 3: Results of the analysis of some pharmaceutical preparations containing Xylometazoline

Pharmaceutical	Order of derivative	Mode of calculation	Drug Conc. µg.ml^{-1}		Rec%
			Taken	Found	
Otrivin/Drops 1mg GlaxoSmithKline Egypt	First	Peak to baseline of 268nm	10	10	100
			20	19	95
DECOZAL/ Drops 1mg AMMAN PHARMACEUTICAL INDUSTRIES Jordan		Peak to baseline of 292nm	10	10	100
			20	19	95

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