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Research Article

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RP-HPLC Method of the Estimation of Xylometazoline hydrochloride in Nasal Spray Solution

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Abstract A simple, specific, precise and accurate reverse phase HPLC method has been developed for the determination of nasal decongestive, Xylometazoline hydrochloride in nasal spray solution. The chromatographic separation was achieved on column C18, 150 mmx4.6 mm, 3.5 µm, using PDA detector. The mobile phase was acetonitrile and phosphate buffer pH 3.0 (60:40 v:v), at flow rate 1.0 mL/min, detected at 220 nm, at 35ºC temperature for column. The retention times was at about 1.5minutes.The method was validated according to the ICH guidelines with respect to specificity, linearity (r^2 =0.999), accuracy (98 to 103%), precision (RSD<2%).

Keywords Xylometazoline hydrochloride, Nasal Spray, RP- HPLC, Validation

Introduction

Xylometazoline hydrochloride, imidazole derivative, alpha - adrenergic sympathomimetic. It has a vasoconstrictive effect and reduces mucosal swelling. It is used to treat a stuffy nose that is caused by allergies, colds, or sinus irritation. The onset of action usually occurs between 5 and 10 minutes, and is manifested by easier breathing through the nose due to decreased mucosal swelling and improved secretion. The action lasts between 6 and 8 hours [1, 2, 3]. The aim of this papaer is to to represent simple, specific, precise, accurate [4] and low cost RP-HPLC method for determination of xylomethazoline hydrochloride in Nasal Spray solution, with very short chromatographic run time (less than 5 minutes). Also, results for two two marketing samples correspond to ICH 3AQ11a. Specification and Control Tests on the Finished Products [5].

Materials and Methods

Instrumentation HPLC system (Agilent technology) consisting of gradient pump, Auto sampler, column oven and photodiode array detector (PDA, Agilent technology) was employed for analysis. Chromatographic data was acquired using chemstation software.

Reagents and Materials

Xylomettazoline hydrochloride was supplied by USP. Acetonitrile (HPLC, Semikem), Potassiumdihydrogen phosphate and Phosphoric acid (Merck) and Milli-Q Water.

Chromatographic condition

Column C18, 150 mmx4.6 mm, 5 µmw as used as a stationary phase. The mobile phase consisting acetonitrile and phosphate buffer pH 3.0, in the ratio of 60:40 (v/v). The flow rate of the mobile phase was 1.0 mL/min. Detector signal was monitored at a wavelength of 220 nm. The column temperature was kept 35°C and injection volume was 10 µl.

Preparation of standard solutions

The standard stock solution was prepared by transferring 10 mg each of Xylometazoline hydrochloride in a 25 ml volumetric flask. Solvent was water. From this solution was diluted to obtain final standard concentration of Xylometazoline hydrochloride from 18mg/L to 112mg/L, respectively.

Preparation of sample solutions (contains 0.1% of Xylometazoline hydrochloride)

Weigh about 0.8 ml of nasal spray solution in a 10 ml flask, and dilute with water to volume.

Validation of the Proposed Method

The proposed method was validated according to the International Conference on Harmonization (ICH) guidelines [4].

System suitability

The system suitability test as per method should be performed and checked before performing any parameter (% RSD).

Linearity and range

A standard linearity solution was prepared to different concentration of 22.5 %, 50 %, 75 %, 100 % and 140 % of the target concentration.

Method precision (Repeatability)

Five solutions were prepared from Xylometazoline hydrochloride. Then they were injected by one analyst and analysed on same day.

Accuracy

The accuracy of the method was carried out at three levels in the range of 75-140% of the working concentration of sample. Calculated amount of Xylometazoline hydrochloride working standards were added in placebo containing volumetric flasks to, prepare 75%, 100% and 140% level of the working concentration.

Specificity

A blank preparation, standard preparation and placebo preparation were prepared and injected.

Result and Discussion

The method has provided very fast evaluation for Xylometazoline hydrochloride. Separation was obtained by using Column C18, 150 mmx4.6 mm, 5 µm as a stationary phase at 35°C temperature and using a mobile phase consisting acetonitrile and phosphate buffer pH 3.0, in the ratio of 60:40 (v/v), at a flow rate 1.0 ml/min and wavelength for detection was 220nm. Under these optimized conditions, the analyte peaks were well resolved and free from tailing.

The tailing factors was0.98and retention time at 1.5 minutes. Specificity of chromatograms shown in figure 1 and system suitability was established by injected standard solution and results are shown in table 1.

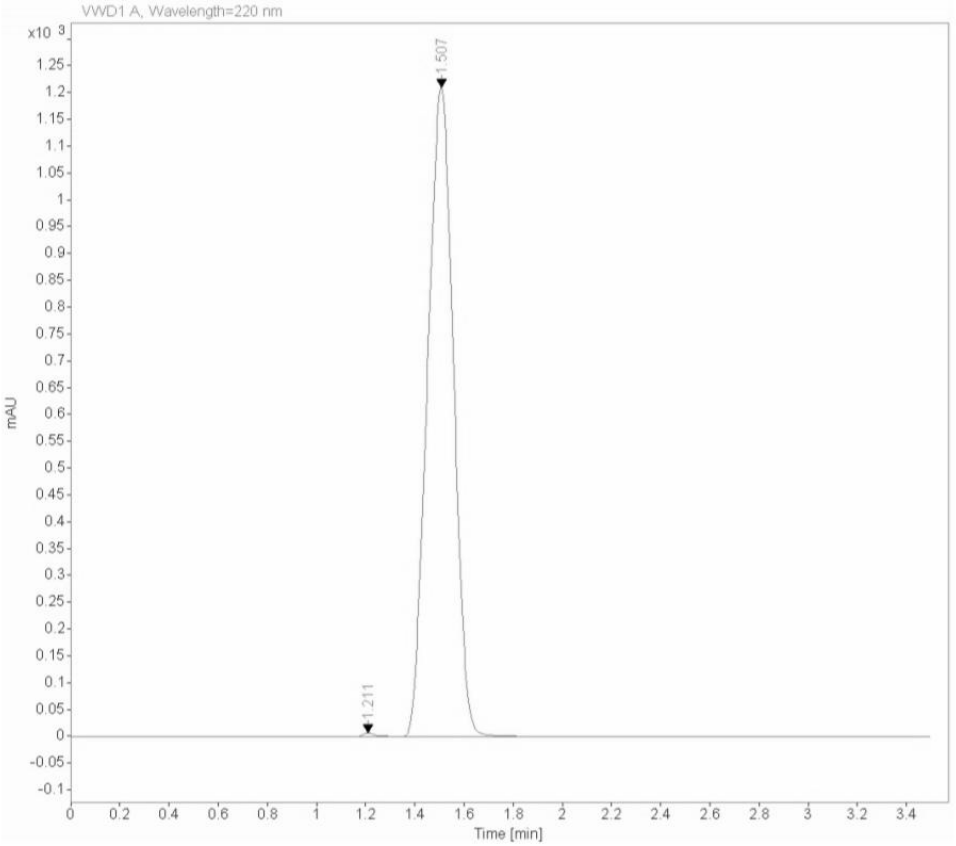

Figure 1: Chromatogram of Xylometazoline hydrochloride peak

Table 1: System suitability parameters

Component	Area	Symmetry	Plate
Xylometazoline hydrochloride	8771	0.98	7038

Specificity of the chromatograms was checked for the appearance of any extra peaks. No chromatographic interference from nasal solution excipients was found.

Linearity of Xylometazoline hydrochloride showed linearity in the range of 18- 112mg/L. The data for the peak area against the concentration were treated by linear regression analysis and the correlation coefficient value obtained was 0.999.

The accuracy was expressed as the percentage of analyte recovered by the assay method. It was confirmed from results that the method is accurate (Table 2).

Table 2: Accuracy data

Level (%)	RSD	
	Xylometazoline hydrochloride	Mean RSD (%)
75	103.1	
100	103.2	101.4
140	97.8	

In Precision, the relative standard deviations (RSD) was 0.7, which are well within the acceptable limit of 2.0%. Results are presented in table 3.

Table 3: Precision recovery data

Sample	Recovery (%)
1	99.1
2	100.4
3	99.6
4	100.7
5	100.2
Average (%)	**100.0**
RSD (%)	**0.7**

Results content for two samples of finished product are presented in table 4.

Table 4: Results for finished products

Sample	Xylometazoline hydrochloride	
	Found (mg/ml)	Found (%)
Sample 1 (0.1% nasal spray solution)	1.00	100.3%

Conclusion

Proposed RP-HPLC method is specific, accurate and precise for the determination of Xylometazoline hydrochloride in pharmaceutical nasal spray solution. The newly developed method is simple and cost effective as it uses simple mobile phase. The evaluation is in five minutes only. The method was validated as per ICH guidelines. All other parameters such as specificity, linearity, precision and accuracy, passes the criteria set forth by ICH guidelines. The described method is suitable for routine analysis and quality control.

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