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CHROMATOGRAPHIC STUDIES OF MULTICOMPONENT VAGINAL SUPPOSITORY

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ABSTRACT

The article presents the results of quantitative research and identification of active- pharmaceutical ingredients (APIs) vaginal suppository. To conduct the study using high performance liquid chromatography (HPLC), as it is the most important method of analysis Pharmacopeia substances and medications (drugs).HPLC carried out on the chromatogram using the parameters of retention components. The quantitative content of API studied by comparing the peak areas of the standard and analyte substances obtained under identical conditions.

Keywords: Chromatographic Parameters, Quantitative Content, The active pharmaceutical ingredient, Delay time, Peaks.

INTRODUCTION

Chromatography - the most commonly used analytical method which has been successfully used in research and clinical purposes in the fields of pharmaceuticals, biology and medicine.

The most important chromatographic parameters allowing to evaluate the efficacy and selectivity of the column and the degree of separation of different substances are: the coefficient of capacitance retention ratio, number of theoretical plates, the height equivalent to a theoretical plate, selectivity coefficient and the coefficient of separation [1, 2 3]. For quantitative analysis of the chromatogram of the detector is transmitted to an electronic device that converts it into digital form, or a chart recorder with a tape. Quantitative analysis performed on the peak height or area, as these parameters are proportional to the concentration or amount of a substance in a chromatographic zone [4, 5].

MATERIALS AND METHODS

The study involved vaginal suppositories. Quantitative determination of the API in vaginal suppositories performed by liquid chromatography in accordance with the requirements of the European Pharmacopoeia 2.2.29, 2.2.46.Methods of preparation of the buffer solution (pH 3.9)4.35 g of potassium phosphate dissolved in 1000 ml of ultrapure water and the resulting solution filtered through a membrane filter with a pore diameter of 0.45 microns.

The method of preparation of the test solution:

Accurately weighed, equivalent to 1 suppository placed in a conical flask of 100 ml of methanol, added 40 ml P is heated for 15 minutes at a temperature of 600 C, and then allowed to stand in an ultrasonic bath for 15 minutes. The resulting solution cooled in an ice bath to freeze bases and filtered through filter paper (blue ribbon) in a volumetric flask of 200 ml. Procedure repeated twice, collecting the filtrate in the same flask. The filter washed with methanol, the solution volume adjusted to the mark with methanol and stirred.10.0 ml of this solution placed in a 25 ml volumetric flask, diluted to volume and mixed solvent.

The method of preparation of the reference solution:

40 mg (accurately weighed) of the standard sample (SB), metronidazole , 160 mg (accurately weighed) of the standard sample (SB) Clotrimazole , 10 mg (accurately weighed) of the standard sample (SB) and progesterone (24 mg, accurately weighed) of the standard sample (SB) ibuproffen placed in a volumetric flask of 50

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ml, dissolved in 30 ml of methanol , the solution volume adjusted with the same solvent to the mark and mixed. 5.0 ml of this solution placed in a volumetric flask of 20 ml volume adjusted to the mark with the solvent solution and stirred.Before analyzing equilibrated in the chromatographic system administered 20 microliters of the reference solution, to give at least 5 repeat chromatograms.

The contents of metronidazole, clotrimazole, progesterone, ibuprofen at 1 suppository, in milligrams calculated from the formula (1):

$$X = \frac{S_1 \times m_0 \times 5 \times 200 \times 25 \times P \times b}{S_0 \times 100 \times 20 \times m_1 \times 10 \times 100}, \quad (1)$$

Where:

S1 - an average value of the peak areas of metronidazole, clotrimazole, progesterone, ibuprofen, calculated from the chromatogram of the test solution, respectively;

S0 - average value of the peak areas of metronidazole, clotrimazole, progesterone, ibuprofen, calculated from the chromatogram of the reference solution, respectively;

m0 - sample weight standard sample metronidazole, clotrimazole, progesterone, ibuprofen, respectively, in mg;

b - the average weight of a suppository, in grams;

m1 - mass of sample equivalent to 1 suppositories, in grams;

F - content of metronidazole, clotrimazole, progesterone, ibuprofen standard sample metronidazole, clotrimazole, ibuprofen, respectively, in%.

The contents of metronidazole, clotrimazole, progesterone, ibuprofen in 1 suppositories Y,% of the declared amount calculated by the formula (2):

$$Y = \frac{X \times 100}{LC}, \quad (2)$$

Where:

X - content metronidazole, clotrimazole, progesterone, ibuprofen in 1 suppositories, respectively mg;

LC - the claimed amount of metronidazole, clotrimazole, progesterone, ibuprofen, listed in the "Ingredients", respectively, in mg / supp [5- 7].

RESULTS AND DISCUSSION

Chromatographic studies allowed determining the time delay peaks all API vaginal suppository. Approximate peaks retention times were metronidazole - 4, 0 min, ibuprofen - 15, 3 min, progesterone - 22, 7 min and clotrimazole - 27 min.

Chromatogram obtained with reference solution allows determining the time delay peaks APIs and comparing them with the time delay peaks API test solution. Table. 1 shows the results of studies quantifying the API.

Table 1:Quantitative content API in preparation

Ingredients	Content, mg	Defined		
		mg / g	%	Metrological characteristics
Metronidazole	100 mg	92,3 91,0 92,3 90,9 mean value. 91,6	92,3 91,0 92,3 90,9	X = 91,6 $S_{(X)}$ = 0,78 $S\bar{x}$ = 0,34 ε = ± 1,05 $X \pm S\bar{x}$ = 91,6 ± 1,05
Clotrimazole	400 mg	425,7 410,9 425,4 410,7 mean value 418,2	106,4 102,7 106,4 102,7	X = 104,55 $S_{(X)}$ = 2,13 $S\bar{x}$ = 0,95 ε = ± 2,54 $X \pm S\bar{x}$ = 104,55 ± 2,54
Progesterone	25 mg	26,9 26,2 27,1 26,4 mean value 26,7	107,7 104,9 108,5 105,6	X = 106,6 $S_{(X)}$ = 1,7 $S\bar{x}$ = 0,76 ε = ± 1,98 $X \pm S\bar{x}$ = 106,6 ±1,98
Ibuprofen	60 mg	56,2 54,9 56,4 55,1 mean value 55,6	93,7 91,5 94,0 91,8	X = 92,75 $S_{(X)}$ = 1,28 $S\bar{x}$ = 0,57 ε = ± 1,71 $X \pm S\bar{x}$ = 92,75 ± 1,71

From the above results show that the content of metronidazole in a 1 suppositories were 91.6 mg (normal 90.0 - 110.0 mg), 418 mg - clotrimazole (at a rate of 360.0 - 440.0 mg), 26.65 mg of progesterone (at a rate of 22.5 - 27.5 mg), ibuprofen 55.65 mg (normal 54.0 - 66.0 mg).

Figure 1 chromatogram of the test solution

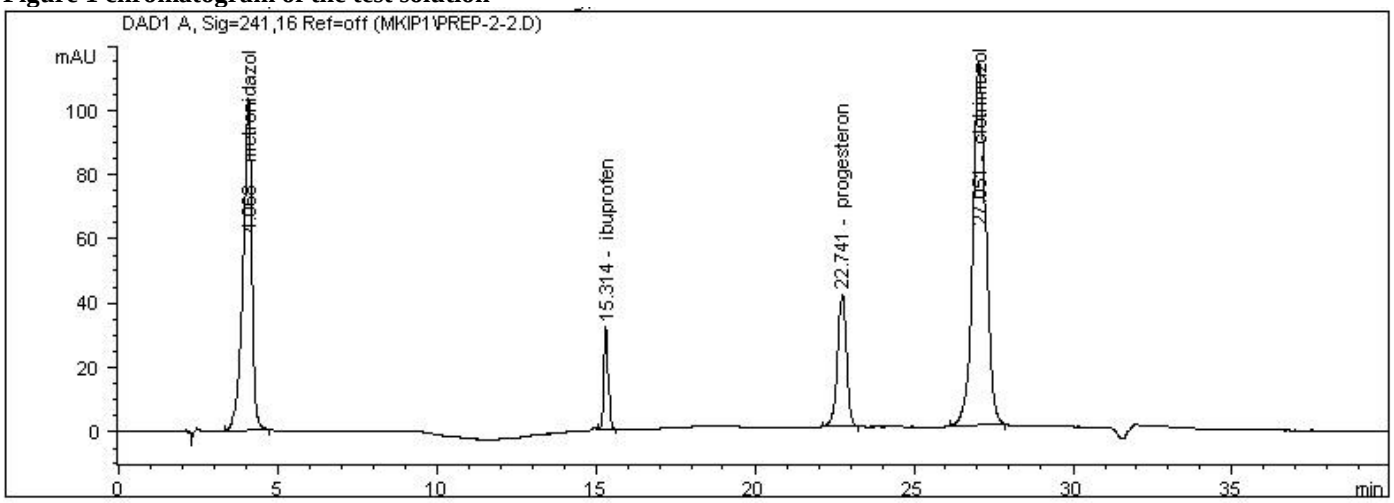
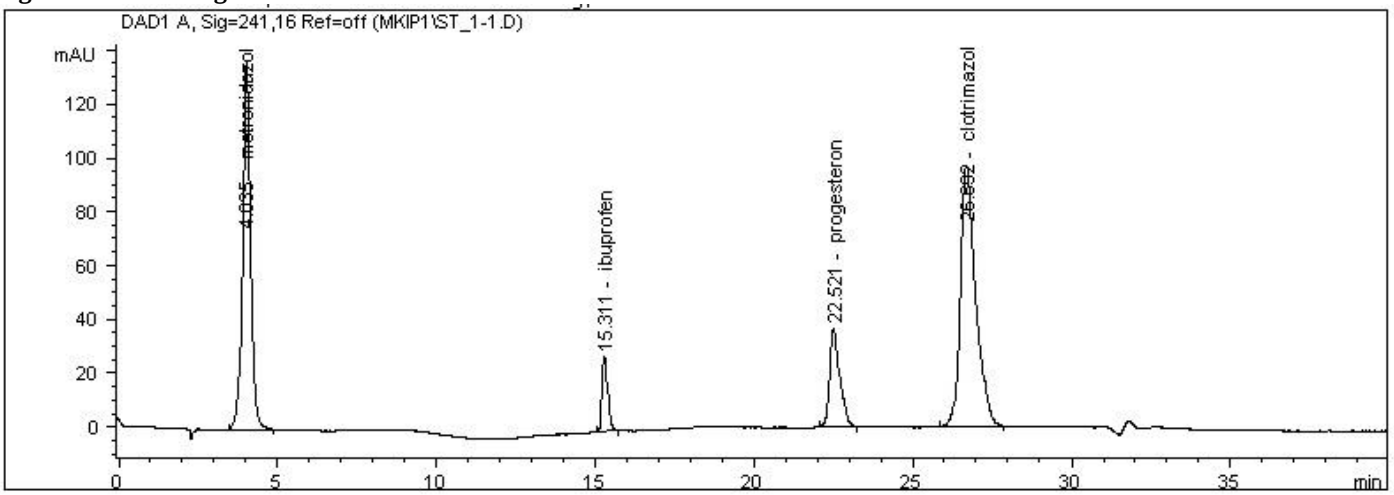


Figure 2 Chromatogram of the reference solution



CONCLUSION

Chromatographic investigation carried out by HPLC showed that the chromatography conditions described provide sufficient selectivity and separation efficiency.Retention time peaks of metronidazole, clotrimazole, ibuprofen and progesterone test solution and the reference solution does not exceed 2%. Identified by chromatographic methods of research needed to develop an optimal composition of a sound technology vaginal suppository and check its quality.

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