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Development Of A Method For Identification And Validation Of A Drug Model Mixture Usingthin-Layer Chromatography

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Abstract

The article presents studies on the development of a method for identifying ibuprofen, papaverine and dibazole in a model drug mixture using the TLC method. According to the results of chromatography "System", "Phase mobility" of the studied substances in individual solvents in different ratios, it was found that the system of chloroform – ethanol – ammonia conc. (1:1:0.5) is more suitable and provides optimal mobility. The polarity of the combined eluents was estimated by the value of the dielectric constant and the volume fraction of individual solvents included in the PF. It is shown that the developed end-to-end method for assessing the quality of the main active substances in the model medicinal mixture can be used in combined dosage forms. Validation parameters such as the specificity and reproducibility of the developed TLC technique were also determined, studied on various chromatographic plates, and its reliability was evaluated based on the obtained Rf parameters. The results of validation of the TLC technique revealed its sensitivity, selectivity, and reproducibility, which will be very important (according to the requirements of the international harmonized standards for quality control ICH) for the analysis of the finished dosage form of antihypertensive action containing ibuprofen, papaverine, and dibazole.

Keywords: Papaverine, dibazole, ibuprofen, thin-layer chromatography, validation, selectivity, reproducibility.

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1. Introduction

Currently, the most relevant issues of rational use of medicinal products are the possibility of evaluating the effectiveness, safety and quality of drugs in circulation throughout their entire "life cycle": from development, quality criteria for pre-registration studies, examination procedures to entry into wide clinical practice and the period of stay on the pharmaceutical market [1-3]. For this purpose, it is necessary to introduce optimal, scientifically based approaches that provide the best benefits and lowest costs for effective, rational, effective and safe therapy with combined drugs [4-6].

Providing the population with high-quality domestic medicines with an innovative modern approach is one of the promising areas of modern pharmacotherapy. Of particular interest are the combined known drugs in rational dosage forms. In this case, there are already complete characteristics of the dependence of effects on a wide range of doses for each drug from the studied combinations [7,8]. It is known that about 30% of people in the world take vasodilators, and 50% of these patients are older than 50 years. The selection of a combination of vasodilating and nonsteroidal anti-inflammatory drugs in rational dosage forms is relevant. Currentlyш, rectal dosage forms in the form of suppositories are becoming increasingly popular in pharmaceutical practice. Their popularity and prospects can be assessed by the fact that medicinal substances, bypassing the gastrointestinal tract, enter directly into the general circulation. They are superior in terms of the rate of absorption, reduction of the degree of allergiziruyushee action, simplicity and painlessness of administration, as well as the absence of danger of introducing infections [9-11]. Thin-layer chromatography (TLC) is widely used to determine the main active substances in multicomponent dosage forms [12-15]

Objective: To develop a method for identification of papaverine and dibazole ibuprofen in a model drug mixture using TLC as a cross-cutting technique for quality assessment in mild combined-action dosage forms.

2. Methods

Model mixture ibuprofen-0.2., papaverine-0.02, dibazole-0.02. To determine the optimal separation conditions used, SILUFOL “SILUFOLUV-254, plates and organic solvents were used. Also developers are iodine vapors, Dragendorf reagent and its modification according to Munier, 5% FeCl3 solution. TLC methodology was validated according to ICH requirements.

3. Results And Discussion

Authenticity was determined by TLC. A solution of the model mixture was applied to the start line using a calibrated capillary for 0.1 ml. Solutions of papaverine, dibazole, and ibuprofen were applied in the same amounts at concentrations corresponding to the content of the model mixture solution. The solvent system chloroform-ethanol in the ratio (8:2), ether-dimethylformamide-benzene in the ratio (4:4:2) was used as the mobile phase. Chloroform-ethanol-ammonia conc. (1:1: 0.5)

To select the optimal composition of the mobile phase (PF), the chromatographic mobility of the mixture substance was studied in individual and combined solvents of different polarities, as well as in systems with different contents of acid and alkaline modifiers (acetic acid and 25% ammonia solution). The obtained chromatograms were dried in a stream of air at room temperature, and the values of Rf were developed and calculated.

Chromatography was performed in chambers saturated with iodine vapors, kept in a solvent. As the required Rf plates were reached, they were removed and dried.

Some metrological characteristics and validation parameters of the qualitative analysis of the model drug mixture were calculated using the TLC method.

The reproducibility of the developed TLC technique was evaluated using various chromatographic plates. The reliability of the method was determined by the obtained values of the retention coefficient (Rf). The results of the TLC method reproducibility study for the model mixture are presented in Table 1.

Table 1

Results of studying the reproducibility of the model mixture

№	Plate No	Rf I +P+ D
1	Silica gel KSK	0.58 0.55 0.50
2	"SilufolUF-254 "(Czech Republic)	0.62 0.50 0.68

3	"SilufolUF-254 "(Mersk)	0.65 0.68 0.70
4	"Sorbfil UV" Russia	0.73 0.68 0.76

It is established that the obtained Rf values when using Silufol UV-254 plates (Mers) meet the requirements of the State Pharmacopoeia of the Republic of Uzbekistan (GF Uz), which confirms the high reproducibility of the developed technique.

Table 2 shows the results of selecting optimal conditions for the separation of papaverine, dibazole and ibuprofen for their chromatographic behavior on the above-mentioned sorbents using mobile phases.

Table 2.

Results of selecting optimal separation conditions

Mobile Phase	Rf		
Substance Study	P	D	And
Ether-dimethylformamide-Benzene	0.80	0.75	0.80
Chloraform-ethanol	0.78	0.67	0.77
chloroform-ethanol-ammonia conc. (1:1:0,5)	0,65	0,68	0,70

Determination of the sensitivity of the TLC method consisted in the preparation of aqueous solutions of the studied samples in variousых концентрациconcentrations and in chromatography in the chloroform-ethanol-ammonia system conc. (1:1:0.5). The results of measuring the sensitivity and detection limit are presented in Tableпределения чувствительности и предела обнаружения представлены в табл.

Table 3

Determination of the sensitivity and detection limit of test substances with various developing reagents

Amount of substance taken for analysis mcg	UV-254	Iodine Vapor	$CuSO_4$	$FeCl_3$
100	+	+	+	+
50	+	+	+	+

10	+	+	+	-
5	+	+	-	-
1	–	+	–	–
0,1	–	–	–	–

From the presented table 3, it can be seen that when analyzing the test substances in a model drug mixture, the detection limit is in the range from 100 to 5 micrograms, and the most optimal developing reagents are viewing in UV light and iodine vapors.

The specificity of TLC methods was studied by chromatography of working and standard samples. For this purpose, we performed a series of chromatographic determinations under the developed chromatography conditions.

The reproducibility of the developed TLC technique was studied on various chromatographic plates andf its reliability was evaluated based on the obtained Rf parameters. Results of studying the reproducibility of TLC methods the obtained indicators Rf meet the requirements of GF XI, GF RUz. The results of validation of the TLC technique revealed its sensitivity, selectivity, and reproducibility, which is very important for analyzing a cross-cutting technique for use in the finished dosage form. According to the results of chromatography "System", "Phase mobility" of the studied substances in individual solvents in different ratios, it was found that the chloroform – ethanol – ammonia conc. (1:1:0.5) system is more suitable and provides optimal mobility, where Rf are indicated in Table 2.

The polarity of the combined eluents was estimated by the value of the dielectric constant and the volume fraction of individual solvents included in the PF. It was found that in the presence of an ethanol solution, the chromatographic mobility of substances increases, and the introduction of ammonia into the PF composition leads to its decrease for both systems. Then, chromatography in systems with different contents of acid and alkaline reagents was evaluated as the average of five parallel determinations. Mobility of substances during chromatography in combined eluents on Silufol UV254 plates (Merck) in the presence of acid modifiera.

The obtained Rf parameters meet the requirements of GF XI and GF RUz, and confirm the reproducibility of the developed TLC technique. Today, Silufol UV254 plates are more affordable and do not require much time to prepare the chromatographic plate, which significantly reduces the analysis time. The results of TLC validation revealed its sensitivity, selectivity, and reproducibility, which will be very important (according to the requirements of the international harmonized standards for quality control ICH) for the analysis of the finished dosage form of antihypertensive action containing ibuprufen, papaverine, and dibazole.

4. Conclusions

Thus, a method was developed for identifying the studied drug model mixture containing ibuprofen, papaverine and dibazole, as well as determining the sensitivity and limit of their detection using various reagents, and it was found that the optimal developing reagents are viewing in UV light and iodine vapor.

Validation parameters such as the specificity and reproducibility of the developed TLC technique were also determined, studied on various chromatographic plates, andf its reliability was evaluated based on the obtained Rf parameters. The results of validation of the TLC technique allowed us to reveal its sensitivity, specificity, selectivity, and reproducibility, which is very important for using the developed end-to-end technique for analysis in a ready-made drug form of antihypertensive action.

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