

МІНІСТЕРСТВО ОХОРОНИ ЗДОРОВ'Я УКРАЇНИ  
НАЦІОНАЛЬНИЙ ФАРМАЦЕВТИЧНИЙ УНІВЕРСИТЕТ

**TOPICAL ISSUES OF NEW MEDICINES  
DEVELOPMENT**

МАТЕРІАЛИ  
XXVI МІЖНАРОДНОЇ НАУКОВО-ПРАКТИЧНОЇ  
КОНФЕРЕНЦІЇ МОЛОДИХ УЧЕНИХ ТА СТУДЕНТІВ

10-12 квітня 2019 року  
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Харків  
НФаУ  
2019

nm. The possibility of isolating naphazoline by the method of liquid – liquid extraction from smears of the nasal mucosa and its qualitative determination was studied. After application of the mucous membrane 1 – 3 drops of naphazoline solution after 1 minute, 0.5 hours and 1 hour, a smear was taken with a damp cotton swab. A cotton swab was placed in a test tube containing 5 ml. purified water at different pH values of the medium (buffer solution). Insisted with rewrite for 10 minutes. The liquid was poured into a separatory funnel. Liquid extraction was performed with 5 ml of an organic solvent (the operation was repeated 3 times). The following solvents were used for isolation: chloroform, ethyl acetate, hexane. The extracts were combined, the emulsion was destroyed by centrifugation. The anhydrous sodium sulfate was passed through sodium into porcelain cups and evaporated in a water bath to dryness. Further spectral characteristics were taken only at a wavelength of 270 nm., As indicated above. The results of the dependence of optical density on the time of application and the amount of naphazoline applied to the nasal mucosa are presented in table 2.

Table 2

The dependence of optical density on the time of application and the amount of naphazoline applied to the nasal mucosa

The time after which the determination was carried out	The amount of naphazoline installed on the nasal mucosa	
	1 drop	3 drops
	Absorption	
1 minute	0,61	0,84
0,5 hour	0,35	0,51
1 hour	0,17	0,29

As can be seen from table 2. Naphazoline can be determined using nasal smears from the nose using UV spectrophotometry for one hour.

**Conclusions.** Isolation of the analyte from nasal swabs should be performed using liquid-liquid extraction with chloroform. Conditions have been developed for liquid – liquid extraction of naphazoline isolated from nasal smears and qualitative determination by UV spectrophotometry. It is established that UV spectroscopy allows identifying the component of the drug naphazoline in washings from the nose for 1 hour.

#### DEVELOPMENT OF THE METHODS OF CINNARIZINE DETECTION AND QUANTITATIVE DETERMINATION SUITABLE FOR THE CHEMICAL-TOXICOLOGICAL ANALYSIS

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**Introduction.** Cinnarizine (1-benzhydryl-4-[(E)-3-phenylprop-2-enyl]piperazine) is a piperazine derivative, is a widely prescribed medication for the treatment of vestibular disorders and motion sickness (nausea, vomiting), vertigo, Meniere’s disease. Some cases of acute intoxications caused by cinnarizine overdose were reported in the literature. Maximum serum cinnarizine level after the drug ingestion was of 7,407 ng/ml. The most of bioanalytical methods for cinnarizine determination are based on using HPLC and GLC.

**Aim.** To develop sensitive and accessible methods for cinnarizine detection and quantitative determination with help of thin layer chromatography (TLC) and UV-spectrophotometry.

**Materials and methods.**  $R_f$  values of cinnarizine in 10 mobile phases including those recommended by The International Association of Forensic Toxicologists for TLC drug screening for 4 types of chromatographic plates (plates manufactured in Estonia with KSKG sorbent, Sorbfil, Silufol UV-254, Merk) were determined. The UV-spectrum of cinnarizine in 0.1 M hydrochloric acid solution was measured over 215–380 nm wavelength range. Stock solution (SS) (20 µg/ml) and 10 working standard solutions (WSS) (1.0; 2.0; 4.0; 6.0; 8.0; 10.0; 12.0; 14.0; 16.0 and 18.0 µg/ml) of the drug were prepared.

**Results and discussion.** Two mobile phases of chloroform – acetone (80:20) ( $R_f=0.36$ ) and methanol – *n*-butanol (60:40) ( $R_f=0.84$ ) (or methanol – 25% ammonia (100:1.5) ( $R_f=0.78$ ), or toluene – acetone – ethanol – 25% ammonia (45:45:7.5:2.5) ( $R_f=0.87$ ), or ethyl acetate – acetone – 25% ammonia (50:45:4) ( $R_f=0.78$ )) had a low correlation of  $R_f$  values (are given for Merk plates). Absorption maxima were detected at wavelength of  $228\pm 2$  and  $254\pm 2$  nm. The calibration curve was described by the following equation:  $y=(0.0566\pm 0.0008)x+(0.070\pm 0.008)$  ( $r=0.9996$ ), LOD and LOQ values were of  $0.2\ \mu\text{g/ml}$  and  $0.7\ \mu\text{g/ml}$ , respectively. The linearity of the calibration curve was within the range of cinnarizine concentrations from 1.0 to  $18\ \mu\text{g/ml}$ .

**Conclusions.** The developed methods of cinnarizine detection and quantitative determination using TLC and UV spectrophotometry are sensitive and selective enough for chemical-toxicological analysis.

## UV-SPECTROPHOTOMETRIC DETERMINATION OF TINIDAZOLE IN ACID MEDIUM

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**Introduction.** Tinidazole – 1-[2-(ethylsulfonyl)ethyl]-2-methyl-5-nitro-1H-imidazole – is the derivative of 5-nitroimidazole and the medicine from the group of antiprotozoal compounds widely used for treatment of infectious diseases.

**Aim.** To develop UV-spectrophotometric procedure of tinidazole quantification using 0.1 M HCl solution as a solvent and carry out step-by-step validation of the developed procedure to choose the optimal variant for further application.

**Materials and methods.** Tinidazole was of pharmacopoeial purity. All spectrophotometric measurements were carried out using a single beam UV/VIS spectrophotometer SPEKOL®1500 (Analytik Jena AG, Germany).

The stock and model solutions were prepared by dissolving tinidazole in 0.1 M hydrochloric acid solution.

The absorbance of the solutions was measured 3 times with randomization of cell position. 0.1 M hydrochloric acid solution was used as a compensation solution.

**Results and discussion.** UV-spectrum of the tinidazole solution in 0.1 M HCl has the absorption maximum at  $\lambda_{\text{max}} = 277$  nm. The value of specific absorbance has been calculated for the concentration range of 5 – 35  $\mu\text{g/mL}$  and  $A_{1\text{cm}}^{1\%} = 253$ .

Validation of the developed procedure has been carried out by model solutions in the variants of the method of calibration curve and method of standard. Such validation parameters as in process stability ( $\delta^{\text{model stability}} = 0.48\%$ ), linearity/calibration model ( $D = 25\% - 175\%$  in normalized coordinates,  $b^{\text{model}} = 0.990\pm 0.020$ ,  $a^{\text{model}} = -0.966\pm 1.647$ ,  $RSD_0^{\text{model}} = 1.571\%$  and  $R_c^{\text{model}} = 0.9994$ ), accuracy ( $\bar{Z}^{\text{model}} = 100.74\%$ ,  $\bar{RR}^{\text{model}} = 99.95\%$ ) and precision ( $\Delta_Z^{\text{model}} = 4.54\%$ ,  $\Delta_{RR}^{\text{model}} = 3.62\%$ ) have been estimated by model solutions.

The total results of validation allow to point to the conclusion about acceptable linearity, accuracy and precision of the developed UV-spectrophotometric procedure of tinidazole quantitative determination in the variants of the method of calibration curve and method of standard.

**Conclusions.** A new procedure of tinidazole quantitative determination by the method of UV-spectrophotometry has been developed using 0.1 M HCl solution as a solvent; its acceptability for application has been shown.