показало, що в найбільшій мірі зазначена речовина екстрагується хлороформом при зміні pH середовища від 2 до 12 (ступінь екстракції складала від 82 до 96%, для діетилового етеру ступінь екстракції при вказаному значенні pH не перевищувала 78%).

В ході розробки методик виділення фенігідину з сечі та крові було встановлено, що одержана хлороформна витяжка не потребує додаткової хроматографічної очистки. Застосовані нами кольорові реакції, методи ТШХ та УФ-спектрофотометрії виявилися досить чутливими для виявлення досліджуваних нами меж концентрацій фенігідину в біологічних рідинах.

Як видно з результатів кількісного визначення фенігідину, виділеного з крові, а також з підкисленої та підлуженої сечі, за допомогою запропонованої методики рідинно-рідинної екстракції з лужного середовища (pH 8-9) з крові можливо виділити 55±5.2% фенігідину, з підкисленої сечі 64.2±3.3%, з підлуженої сечі – 75±2.5% препарату.

Висновки. Розроблені ефективні методики рідинно-рідинної екстракції фенігідину з біологічних рідин хлороформом з лужного середовища (pH 8-9), що дозволяють виділити з крові 55±5.2%, з підкисленої сечі – 64.2±3.3% та з підлуженої сечі – 75±2.5% фенігідину.

DETERMINATIOM OF IRON(II) IN THE PRESENCE OF THOUSAND-TO-ONE RATIO OF IRON(II) USING BATHOPHENANTHLOLINE

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Introduction. In a publications a few years ago, a method was described for the determination of Fe(II) in the presence of 1.00 mg of Fe(III). Modifications of that bathophenanthroline procedure permit the determination of Fe(II) in the presence of 25.0 mg of Fe(III). The present procedure is quicker, and has a higher molar absorptivity that earlier procedures.

Aim. The results of our research was discovered the possibility of using Fe(III) oxidized with $(NH_4)_2S_2O_8$ in suitable low blanks.

Materials and methods. The sample solution was placed in a beaker and the volume adjusted to 20 cm^3 with water. Ten ml of 10% $NH_4H_2PO_4$ was added and the pH adjusted to 2.00-2.10 with 3M sodium acetate. The solution was transferred to a separatory funnel; 30 cm^3 of the bathopheanthroline solution was used to rinse the beaker and the rinse was added to the separatory funnel. After shaking for a few seconds and allowing the sample to stand for a few minutes, 10 cm^3 of chloroform was added by pipet and the sample was shaken for 45 seconds. The layers were allowed to separate for a few minutes and then the organic layer was drained into a dry 25-cm^3 volumetric flask. The solution was made to volume

Results and discussion. The use of $NH_4H_2PO_4$ makes it possible to complex Fe(III) completely. Sodium acetate is used to adjust the pH 2.00-2.10 because too large a concentration of $NH_4H_2PO_4$ results in the inhibition of the ferrous-bathophenanthroline complex. The use of 10 cm^3 of $NH_4H_2PO_4$ and sodium acetate results in very nearly total complexation of Fe(III) and the usual molar absorptivity of 22,000 for Fe (II).

It is necessary that the ethanol to aqueous concentration be 1:1, or the chloroform layer does not separate readily. In the extraction using 10 cm^3 of chloroform, approximately 10 cm^3 of alcohol and all of the ferrous bathopheanthroline readily separates into the lower organic layer.

The present procedure results in the determination of Fe(II) in Fe(III) in the ratio of 1000:1 with essentially no more difficulty that in the conventional determination of total iron in an applicable matrix.

Conclusions. Results of our research shows that the Fe(III) standard appeared to have approximately 0.25 μg of Fe(II) per mg of Fe(III) and color development of the Fe(II) is independent of time over a 1-to-10 minute time span.

UV-SPECTROPHOTOMETRIC DETERMINATION OF TINIDAZOLE, ORNIDAZOLE AND NIMORAZOLE IN ACID MEDIUM IN THE VARIANT OF THE METHOD OF ADDITIONS

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Introduction. Tinidazole (1-[2-(ethylsulfonyl)ethyl]-2-methyl-5-nitro-1*H*-imidazole), ornidazole (1-chloro-3-(2-methyl-5-nitroimidazol-1-yl)propan-2-ol) and nimorazole (4-[2-(5-nitroimidazol-1-yl)ethyl]morpholine) are the derivatives of 5-nitroimidazole and the medicines from the group of antiprotozoal compounds widely used for treatment of infectious diseases.

Aim. To develop UV-spectrophotometric procedure of tinidazole, ornidazole and nimorazole quantitative determination using 0.1 M HCl solution as a solvent and carry out step-by-step validation of the developed procedure in the variant of the method of additions.

Materials and methods. Tinidazole, ornidazole and nimorazole were 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, and also solution of addition were prepared by dissolving the substances in 0.1 M hydrochloric acid solution.

The absorbance of all 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-spectra of the solutions of tinidazole, ornidazole and nimorazole in 0.1 M HCl have the absorption maximum at 277, 277 and 298 nm respectively. The values of specific absorbance have been calculated and equal 195 (tinidazole, concentration range: 6 – 42 μg/mL), 212 (ornidazole, concentration range: 5 – 35 μg/mL), 159 (nimorazole, concentration range: 7 – 49 μg/mL).

Validation of the developed procedures has been carried out by model solutions in the variant of the method of additions. The analytical range *D* of the methods application is 25 – 175%; the number of concentration levels *g* equals 7 in constant increments of 25%. Such validation parameters as in process stability, linearity/calibration model, precision and accuracy have been estimated by model solutions.

To estimate precision and accuracy the model solutions with and without addition were analysed within 1 run; the concentrations of model solutions without addition were recalculated:

$$X_{ad}^{model} = \frac{C_{ad}^{model} \cdot V_{ad}}{C_{reference}^{model} \cdot V_{m.f}} \cdot 100\%; \quad X_{i,fact}^{model\,MA} = \frac{C_i^{model\,MA}}{C_{reference}^{model}} \cdot 100\%; \quad X_{i,calc}^{model\,MA} = X_{ad}^{model} \cdot \frac{A_i^{model\,MA}}{A_{i+ad}^{model\,MA} - A_i^{model\,MA}}.$$

The values «found/given» $RR_i^{model\,MA}$ were calculated and used to determine the confidence interval $\Delta_{RR}^{model\,MA}$ and the systematic error $\delta^{model\,MA}$ respectively:

$$RR_i^{model\,MA} = \frac{X_{i,calc}^{model\,MA}}{X_{i,fact}^{model\,MA}} \cdot 100\%;$$

$$\Delta_{RR}^{model\,MA} = t(95\%; n-1) \cdot RSD_{RR}^{model\,MA} \le \max \Delta_{As}^{model} = 6.40\%;$$

$$\delta^{model\,MA} = \left| 100 - \overline{RR}^{model\,MA} \right| \le \max \delta^{model} = 2.05\%$$