DEVELOPMENT OF CONDITIONS FOR ORNIDAZOLE DETECTION BY THE METHOD OF THIN-LAYER CHROMATOGRAPHY

Sana El Hardouzi
Scientific supervisors: assoc. prof. Klimenko L. Yu., assoc. prof. Shovkova Z. V., assoc. prof. Kiz O. V.
National University of Pharmacy, Kharkiv, Ukraine
lina_klimenko@nuph.edu.ua

Introduction. Ornidazole is attributed to the group of antiprotozoal medicines and widely used for treatment of infectious diseases; at the same time it is possessed of quite a number of side effects showed by classic symptoms of acute intoxication, especially when interacting with alcohol.

Aim. The research purpose is to develop the conditions of ornidazole identification by the method of chromatography in thin layers of sorbent.

Materials and methods. Ornidazole of pharmacopoeial purity was used in the experiment; its solutions in ethanol with the concentration of 1 mg/mL and 0.1 mg/mL were prepared.

The chromatographic plates Sorbfil® PTLC-PH (silica gel STC-1HP, PETP, silica sol, 8 ÷ 12 μm fraction, 100 μm layer thickness) purchased from IMID LLC (Russia) were used as the thin layers.

Results and discussion. The chromatographic behaviour of ornidazole has been studied in 18 mobile phases: 1. chloroform – acetone (8:2); 2. ethyl acetate; 3. chloroform – methanol (9:1); 4. ethyl acetate – methanol – 25% NH_3 (85:10:5); 5. methanol; 6. methanol – *n*-butanol (6:4); 7. methanol – 25% NH_3 (100:1.5); 8. cyclohexane – toluene – diethylamine (75:15:10); 9. acetone; 10. chloroform – dioxane – acetone – 25% NH_3 (47.5:45:5:2.5); 11. toluene – acetone – ethanol – 25% NH_3 (45:45:7.5:2.5); 12. chloroform – *n*-butanol – 25% NH_3 (70:40:5); 13. chloroform; 14. chloroform – methanol – CH_3COOH conc. (90:10:1); 15. toluene – CH_3COOH conc. (3:1); 16. toluene – methanol – CH_3COOH conc. (9:1:1); 17. ethyl acetate – methanol – CH_3COOH conc. (85:10:2.5); 18. chloroform – methanol (1:1).

When using the mobile phases 3, 5, 8, 9 the investigations were carried out also at the plates processed previously with 0.1 mole/L KOH solution in methanol and then dried at 110°C for 30 min. In the mobile phase 6 the plates were previously processed with 0.1 mole/L NaBr solution.

UV-light, ninhydrin spray, the Dragendorff spray, acidified iodoplatinate solution, the Van Urk reagent and 0.1 mole/L KOH solution in methanol were used for developing the spots of ornidazole at the plates.

Conclusions. The chromatographic mobility of ornidazole has been studied under the conditions of TLC-screening using general and some individual systems of solvents. The reagents for the ornidazole spots development on chromatographic plates have been offered; their sensitivity has been ascertained.

CHOICE OF DEVELOPING REAGENTS FOR EFAVIRENZ ANALYSIS BY THE METHOD OF TLC

Slabiak O. I., Koval Yu. S.
Scientific supervisors: assoc. prof. Ivanchuk I. M., assoc. prof. Klimenko L. Yu., as. prof. Mykytenko O. Ye.
Ivano-Frankivsk National Medical University, Ivano-Frankivsk, Ukraine
National University of Pharmacy, Kharkiv, Ukraine
lina_klimenko@nuph.edu.ua

Introduction. Efavirenz is a non-nucleoside reverse transcriptase inhibitor and attributed to the group of antiretroviral medicines used for treatment of HIV infection. Efavirenz is possessed of quite a number of side effects showed by psychiatric symptoms, including insomnia, nightmares, memory loss, depression, and anxiety. Treatment with efavirenz accompanies with certain neuropsychological symptoms in 50% of cases; its neurotoxicity exceeds other antiretroviral medicines

Aim. To choose the developing reagents and conditions of their application for efavirenz identification by the method of chromatography in thin layers of sorbent.

Materials and methods. Two types of chromatographic plates with a fixed layer of silica gel such as plastic plates with UV-indicator and glass plates without UV-indicator were used as the thin layers. After application of each reagent the plates were examined in visible and UV-light at two wavelengths (254 and 365 nm), then heated for 10 minutes at 110°C and viewed in visible and UV-light one more time.

Results and discussion. More than 50 reagents recommended «Clarke's...» (2011), their modifications, and a number of reagents proposed by us according to the structure of the analyte were used to visualize the spots of efavirenz on chromatographic plates.

The most sensitive developers are:

- concentrated sulphuric acid – light yellow spot;
- the Marquis reagent – light yellow spot;
- the Froehde reagent – brown spot;
- the Mandelin reagent – light brown spot;
- formaldehyde followed by the Mandelin reagent – pink spot;
- the Liebermann reagent – bright yellow spot;
- diphenylcarbazone and $HgSO_4$ – light violet spot;
- ninhydrin – light brown spot;
- acidified iodoplatinate solution – brown spot.

Conclusions. The set of developing reagents for efavirenz analysis by the method of TLC has been offered.

THE CHOICE OF CONDITIONS FOR DETERMINATION OF AMLODIPINE BY METHOD OF DERIVATIVE SPECTROPHOTOMETRY, SUITABLE FOR CHEMICAL-TOXICOLOGICAL ANALYSIS

Souhayl Gaouz, Mamina O. O., Kabachny V. I.
National University of Pharmacy, Kharkov, Ukraine
a_mamina@ukr.net

Introduction. Amlodipine besylate belongs to a group of blockers of calcium channels, derivatives of 1,4-dihydropyridine, is used to treatment of arterial hypertension and vasospastic forms of angina pectoris. Amlodipine reduces peripheral and coronary resistance, improves coronary blood flow, reduces intracellular overload with calcium, suppresses platelet aggregation. According to the literature sources, amlodipine in case of overdose can provoke the development of breast cancer, cause ischemia of the optic nerve. Deadly poisoning with amlodipine may accompany drug overdoses or suicidal cases. Fatal doses for children and adults range from 0,9 to 4,1 mg / kg.

Studies of biological objects in the forensic toxicology departments of the forensic medical examination bureau are conducted in the absence of control experiments. The choice of the method of derivative spectrophotometry for the identification and quantification of drugs in the chemical-toxicological analysis makes it possible to eliminate the influence of the background of impurities and to obtain reliable and reproducible results.

Aim. The aim of this work is the choice of the optimal conditions for the identification and quantitative determination of amlodipine by the method derivative UV spectrophotometry, suitable for chemical-toxicological analysis.

Materials and method. To select the optimal conditions for the analysis by the method of derivative spectrophotometry, UV light absorption spectra of the investigated amlodipine solutions were measured with a spectrophotometer SF-46 in the wavelength range 200-350 nm in a cuvette with a layer thickness of 10 mm. As a solution of comparison was used the corresponding solvent.

The second derivative of the absorption spectra ($d^2A / d\lambda^2$) was calculated using the least-squares polynomial approximation. The calculation of the second derivative for wavelength λ_3, the optical density of amlodipine solutions was measured at wavelengths λ_1, λ_2, λ_3, λ_4, λ_5 with an interval of 4 nm. The obtained values were multiplied by the polynomial coefficients for the corresponding number of points. Identification of the test substance was carried out in the presence of maxima, minima and points of