

Union, the method of viscous liquid chromatography with tandem mass spectrometry is used to identify antibiotics in biosphere objects. This method is highly sensitive, fairly fast and efficient, but very expensive and requires special equipment. Therefore, the use of such analysis is limited.

**Aim.** The aim of this work is to develop a technique for the detection of azithromycin in wastewater by thin-layer chromatography for possible use in ecotoxicological monitoring.

**Materials and methods.** The chromatographic mobility of Azithromycin in thin sorbent layers was studied. The system of solvents providing the best chromatographic mobility of the studied compounds was selected. A technique has been developed for the detection and identification of Azithromycin in water; the optimum mobile phases for chromatography have been selected and Dragendorff's reagent and UV-light have been offered as demonstrators; thanks to them Azithromycin chromatographic zones were clearly detected.

**Results and discussion.** The proposed method of Azithromycin identification can be used for its detection in wastewater and groundwater after preliminary concentration. Using conventional analytical scales and universal chromatography in thin layers of a sorbent it is possible to identify azithromycin with a water concentration of  $\geq 30 \mu\text{g/ml}$  without complex and expensive equipment, such as HPLC or LC/MS/MS.

**Conclusions.** A preparative method for the detection of antibiotic Azithromycin in wastewater by thin-layer chromatography was developed for possible use in ecotoxicological monitoring. Our proposed method is fast, simple, does not require expensive materials and equipment, and is an extremely versatile analytical method that every laboratory can afford. Therefore, the widespread and popular thin-layer chromatography has the right to be another alternative method for the identification of antibiotics in drinking water and to be used for ecotoxicological monitoring.

## DETERMINATION OF PSEUDOEPHEDRINE IN THE MATERIALS OF FORENSIC PHARMACEUTICAL CASES

Zineddaine Ayoub, Grynenko V.V., Bevz O.V.

Scientific supervisor: Bevz N.Yu.

National University of Pharmacy, Kharkiv, Ukraine

ayoubzineddaine02@gmail.com

**Introduction.** Pseudoephedrine ( $\text{C}_{10}\text{H}_{15}\text{NO}$ , mwt. 165.23g/mol), is sympathomimetic drug regulated for medical use. This medication is a known nasal decongestant readily obtained over the counter for temporary relief of stuffy nose and sinus pain/pressure caused by infection (such as the common cold, flu) or other breathing illness such as allergies, bronchitis and hay fever. Pseudoephedrine salts are common active pharmaceutical ingredients in numerous cold medications, commonly sold in a fixed-dose combination with additional active ingredients such as acetaminophen, antihistamines, guaifenesin, dextromethorphan and/or ibuprofen.

Either in bulk form or chemical preparations, pseudoephedrine is strictly regulated for use in chemical, medical and pharmaceutical industries. Legitimate use, however, is diverted by drug trafficking organizations which utilize the compound as the main precursor for clandestine drug production, therefore has captured forensic investigation interest. This diversion has contributed to the continual increase in the use and seizures of a pharmaceutical preparation containing ephedrine/pseudoephedrine worldwide. Despite being listed under 1988 United Nations Convention Against Illicit Traffic in Narcotic Drugs and

Psychotropic Substances which enforces strict regulation in import, export, consumer purchase restriction and behind the counter safe-keeping of pseudoephedrine powder or products, illicit diversion of these precursors is extremely difficult to control. The compound is clandestinely isolated and purified from retail goods to improve batch sizes, yield and purity.

Over 65 million tablets were reported to have been seized around the world between to have been seized last 10 years. As a precursor, the compound is highly coveted to produce some amphetamine-type stimulants through reduction and/or oxidation reactions.

**Aim.** Selection of methods for determining pseudoephedrine in materials of forensic pharmaceutical analysis.

**Materials and methods.** Compilation of data from reports on pseudoephedrine analysis methods suitable for forensic pharmaceutical analysis, mathematical calculations and statistical processing of the results.

**Results and discussion.** From a forensic intelligence perspective, investigation on the origin of the precursor used in the manufacturing process is useful to disrupt the diversion of precursors thus may help drug law enforcement authorities obtain other information of strategic relevance.

Several methods were found in the literature for its quantitation such as titrimetry, spectrophotometry, high-performance thin-layer chromatography, gas chromatography, micellar electrokinetic chromatography, liquid chromatography and capillary electrophoresis.

Chromatographic methods of analysis are the most sensitive and often used in forensic pharmaceutical analysis, because due to separation with the right conditions, it is possible to identify a compound in a mixture, or to determine the original part and new substances synthesized from it. Therefore, we considered possible methods of determining pseudoephedrine in the materials of court cases. The thin-layer chromatography method and the liquid chromatography method turned out to be the most accurate and affordable.

According to the United States Pharmacopoeia, the method of liquid chromatography is used for the determination of the pseudoephedrine hydrochloride. For this method, a liquid chromatograph equipped with a 206-nm detector and a 3.0 mm x 15 cm column is used. The flow rate is about 0.6 ml per minute. The mobile phase is a mixture of triethylamine solution and phosphoric acid solution (90:10). Adjust with phosphoric acid to pH 6.8. Pseudoephedrine hydrochloride solution (1 mg/ml) is separated from Allegra D tablet is filtered through 0.22  $\mu\text{m}$  pore syringe filter and inject 50  $\mu\text{l}$ . Retention time of pseudoephedrine hydrochloride is about 1.85 min. Limit of detection of pseudoephedrine is 5.73  $\mu\text{g/ml}$  and limit of quantitation – 17.33  $\mu\text{g/ml}$ .

Concerning the thin layer chromatography method, aluminum TLC plates pre-coated with silica gel 60F<sub>254</sub> are used and methanol : water : ammonia (9:1:0.1, v/v/v) is applied as a mobile phase; scanning of the plates is carried out at 254 nm. Separate stock standard solution of 5.0 mg/mL pseudoephedrine is prepared in water. The same solvent is used for further dilution in order to prepare the working standard solutions with the concentrations of 2.5 mg/mL pseudoephedrine. Limit of detection is 50.73  $\mu\text{g/ml}$  and limit of quantitation – 153.72  $\mu\text{g/ml}$ . Limit of detection of pseudoephedrine in sample is 0.28  $\mu\text{g/ml}$  and limit of quantitation – 0.84  $\mu\text{g/ml}$ .

**Conclusions.** Clandestine operators can readily extract pseudoephedrine from over-the-counter cold medication tablet either by simple direct extraction or more complicated chemical purification techniques to use as a precursor for illicit drugs. Acid-base extraction can produce a precursor of considerably high purity when compared to pure standards which would make source determination for forensic intelligence challenging. Therefore, the introduction of chromatographic methods for the determination of pseudoephedrine in case materials will allow the identification of pseudoephedrine and products that were obtained illegally.