ПОЗИТИВНИЙ ДОСВІД В ЕКСПЕРТНІЙ ДІЯЛЬНОСТІ
POSITIVE EXPERIENCE IN FORENSIC ACTIVITY

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PIPECURONIUM IDENTIFICATION ACCORDING TO THE SCHEME OF APPLICATION OF ANALYTICAL METHODS RESEARCH DEPENDING ON THEIR SELECTIVITY (SWGDRUG)

The purpose of the article is to comprehensively analyze the theoretical and practical aspects of pipecuronium identification in accordance with the scheme of application of analytical research methods due to their selectivity, and to develop a method of testing a standard sample of pipecuronium by physicochemical methods to confirm substantiated expert report. ***Methodology***. The reliability of the obtained results and conclusions is ensured by the use of a set of general scientific and special research methods. Methods of analysis, synthesis, comparison, generalization allowed to analyze information sources in the field of research, as well as the analytical scheme of a set of physicochemical research methods recommended by SWGDRUG. Approbational analytical study of a standard sample of pipecuronium using experimental methods, analysis, comparison, as well as special physical, chemical, statistical research methods allowed to test a set of physicochemical methods of research of this substance and draw conclusions about the suitability of certain species and outline further research. ***Scientific novelty***. The laboratory of the State Research Forensic Center of the Ministry of Internal Affairs of Ukraine tested the analytical scheme of physicochemical research methods for identification of potent and toxic drugs, recommended by SWGDRUG, and proposed a method of testing a standard sample of pipecuronium by physicochemical methods according to the scheme of analytical methods. due to their selectivity. ***Conclusions***. The SWGDRUG recommendations on the combination of analytical methods are analyzed and the minimum requirements for their application are characterized. It was stressed that in the context of sufficiency for the identification of potent and toxic drugs, in particular heavy substances, expert laboratories, given the physicochemical properties of such substances and analytical equipment available in expert institutions of the country, must determine a combination of methods to ensure reliable results. ***Analytical research***. The possibility of its identification by means of methods: qualitative chemical reactions (with the most available reagents, such as: Marquis, cobalt rhodonite, Dragendorff, Wagner, potassium iodoplatinate, Nessler), IR spectroscopy, mass spectrometry, Liquid chromatography–mass spectrometry with corona discharge detection. The expediency of combining methods is argued, which allows to implement the application of the analytical scheme of research methods recommended by SWGDRUG. It is stated that, given that the use of category B methods is not publicly available, the development of a derivatization process for the study of pipecuronium derivatives by publicly available methods: Gas chromatography-mass spectrometry (GC-MS) and Thin-layer chromatography using different types of sorbents. The results obtained during the approbation of a complex analytical study of pipecuronium according to the recommended international scheme are summarized, and certain proposals are initiated, which can be the basis of research methods of standard sample of pipecuronium by physicochemical methods to confirm the validity, reliability, reproducibility of scientific identification expert report.

Keywords: forensic examination; forensic expert; forensic expert report; SWGDRUG; analytical research

methods used by SWGDRUG; selectivity; analytical scheme of research methods; identification of potent and toxic drugs; hardly volatile compounds; pipecuronium bromide.

Introduction

Forensic experts, while conducting examinations on the study of controlled substances, in order to achieve the proper quality and ensure the scientific validity of their results must adhere to the minimum standards for the identification of chemical compounds.

Improving the quality of such examinations, study and development expertise internationally accepted minimum standards of scientific research takes care of the working group on the analysis of seized drugs (Scientific Working Group for the Analysis of Seized Drugs; SWGDRUG), part of which today includes over twenty legal experts from around the world. Some aspects of this area expressed by scientists and practitioners (Lurie, Marginean, & Rowe, 2006; Tchekhovskoi, Phinney, & Stein, 2017; Naqi, 2019; Wallace, Cascini, De Giovanni, Inserra, Santaroni, & Laura, 2020; Liu, Brettell, Wood, Staretz, & Victoria, 2020, Jun.; Liu, Victoria, Wood, Staretz, & Brettell, 2020, Aug.).

Thanks to the efforts of SWGDRUG as a minimum standard for the identification of chemical compounds, expert laboratories were offered to implement an analytical scheme of research methods (combination of analytical methods) to achieve a scientifically justified result and developed recommendations for their application by selectivity (*Scientific Working Group,* 2019).

However, according to the expert practice of analytical research of hardly volatile compounds, particularly pipecuronium, to apply the recommended combination of analytical research methods in full, given the physicochemical properties of such substances and analytical equipment available in expert institutions of the country, is quite problematic:

certain features of substance identification by thin-layer chromatography (TLC), gas chromatography and ultraviolet-visible spectroscopy;

absence of library mass spectrums (AIPSIN, CAYMAN, SWGDRUG, NIST etc.) and spectrum of infrared spectroscopy;

unfinished methodological approaches and methods of conducting specific chemical reactions.

In addition, it should be noted that the literature and other sources of information contain mostly general data about the physicochemical properties of pipecuronium and its effects on the human body (Kárpáti, & Biró, 1980, p. 346–354; Naguib, & Abdulatif, 1993, p. 556–560; Błażewicz, Fijałek, & Samsel, 2008, p. 191–195; García, Gomes, Santoro, & Kedor-Hackmann, 2008, p. 639–644; *National Center for Biotechnology Information,* 2021) and there is a lack of information that can be used to objectively identify and quantify analysts in accordance with international research approaches and analytical schemes.

The issue of using different methods for the study of hardly volatile compounds was covered by domestic and foreign experts (Mazina, Vaher, Kuhtinskaja, Poryvkina, & Kaljurand, 2015; Blakey, Boyd, Atkinson, Wolf, Slottje, Goodchild, & McGowan, 2016; Breindahl, Kimergård, Andreasen, & Pedersen, 2017; Chia, Ong, Yeo, Ho, Nasir, Tan, Chua, Yap, & Lim, 2019; Yu, Ge, Li, Xie, & Yang, 2019; Ferrari, Arantes, Salum, & Caldas, 2020; Liu, Brettell, Wood, Staretz, & Victoria, 2020, Jun.; Liu, Victoria, Wood, Staretz, & Brettell, 2020, Aug.; Peng, Athukorale, Hu, Cui, & Zhang, 2021); tested in practice the use of publicly available physicochemical methods for the study of pipecuronium (Zamoshets, Barikova, Zelenyi, Kosmina, & Korobchuk, 2021); reviewed instrumental methods of analysis (Studeniak, Voronych, Sukhareva, Fershal, & Bazel, 2014); studied the validation characteristics of hardly volatile compounds, including pipecuronium, using the method of high-performance liquid chromatography, as well as the problem of using different mobile phases for its study (Gazdag, Babják, Kemenes-Bakos, & Görög, 1991; Błażewicz, Fijałek, & Samsel, 2008; García, Gomes, Santoro, & Kedor-Hackmann, 2008).

However, today, despite the interest of scientific community of the world in the outlined issues, in Ukraine the recommended SWGDRUG theoretically constructed analytical methods for the study of hardly volatile compounds for the identification of chemical compounds need to be systematized and tested.

The purpose and objectives of the study

The purpose of the article is to comprehensively analyze the theoretical and practical aspects of pipecuronium identification in accordance with the scheme of application of analytical research methods due to their selectivity, and to develop a testing method a standard sample of pipecuronium by physicochemical methods to confirm justified expert report.

To achieve this goal you need to perform the following tasks:

analyze the SWGDRUG recommendations on the combination of analytical methods, characterize the minimum requirements for their use in the context of sufficiency for the identification of potent and toxic drugs, in particular hardly volatile compounds;

conduct testing of complex analytical research of pipekuronium by the recommended international scheme, including methods of color tests, thin-layer chromatography, spectroscopy in the infrared region of the spectrum (infrared spectroscopy), Gas

chromatography-mass spectrometry (GC-MS), mass spectrometry with direct insertion probe (MS-DIP), high performance liquid chromatography (HPLC);

generalize the obtained results and propose a method of research of a standard sample of pipecuronium by physicochemical methods to confirm the validity, reliability, reproducibility of the results of its identification and sufficiency for the preparation of a scientifically expert report.

Presentation of the main material

Analytical research methods according to the SWGDRUG recommendations for their combination (*Scientific Working Group,* 2019) depending on the level of selectivity (ability to obtain with their use individual information about the characteristics of individual objects that are units of the studied population, to distinguish them) are divided (Naqi, 2019) into three categories (Table 1).

Table 1

Categories of analytical research methods used by SWGDRUG

Category A (Selectivity through Structural Information)	Infrared Spectroscopy
	Mass Spectrometry
	Nuclear Magnetic Resonance Spectroscopy
	Raman Spectroscopy
	X-ray Diffractometry
Category B (Selectivity through Chemical and Physical Characteristics)	Capillary Electrophoresis
	Gas Chromatography
	Ion Mobility Spectrometry
	Liquid Chromatography
	Microcrystalline Tests
	Supercritical Fluid Chromatography
	Thin Layer Chromatography
	Ultraviolet/Visible Spectroscopy
	Macroscopic Examination (Cannabis only)
	Microscopic Examination (Cannabis only)
Category C (Selectivity through General or Class Information)	Color Tests
	Fluorescence Spectroscopy
	Immunoassay
	Melting Point
	Pharmaceutical Identifiers

Analytical methods of category A research allow to obtain information about the structure of the molecule and provide the highest level of selectivity; category B methods – on physicochemical characteristics of samples, providing an average level of selectivity; category C – only about the class of substances, and therefore have the lowest selectivity.

As practice shows, accurate identification of a chemical compound depends on the analytical scheme of proven research methods and the competence of the analytical chemist. SWGDRUG recommends that expert laboratories, using analytical methods, adhere to the following minimum requirements:

in the case of using one of the methods of category A to use at least one other method of category A, B, C;

if research is planned without the use of category A methods, it is mandatory to use at least three different methods, two of which must belong to category B;

if possible, compare the results of analytical measurements of the test sample with the measurement results of the reference (standard) sample; the samples are examined under the same conditions;

a parallel study (or the use of at least two separate samples);

when using combined methods (GC-MS, etc.), they belong to categories (B+A);

it should be remembered that the analytical scheme ensures the achievement of a scientifically sound result only if the application of each of the methods obtained identification results that confirm each other;

the use of a category A method cannot provide a sufficient level of selectivity if the ability to distinguish an analyte (generally a substance or other component of a system to be chemically analyzed, such as a solution, gas mixture or solid component) from similar structural compounds and sample number or the analyte concentration is limited. In this case, the method may form part of the analytical scheme, provided that the limitation is removed by using another method in accordance with the analytical scheme.

Thus, the analytical scheme research methods can be used in such combinations (they are not

exhaustive): Methods category B + A; A + C; C + B + B; C + B + A.

These recommendations are the minimum standards for forensic examination, which will ensure the results for the formulation of a scientifically expert report. However, it should be recognized that these methods may not be sufficient to identify all substances. In this case, expert laboratories must determine which combination of methods will ensure the reliability of the results of analytical studies. (*Scientific Working Group,* 2019).

Pipecuronium, which illustrates the possibility of identifying compounds of unknown nature (chemical compounds) by the scheme of application of analytical methods depending on their selectivity, is one of the representatives of non-depolarizing long-acting neuromuscular blocking agents. A synthetic steroid compound structurally similar to pancuronium and vecuronium that has no hormonal activity. Bis-quaternary structure with piperazine rings attached at positions 2 and 16 of the steroid nucleus (Naguib, & Abdulatif, 1993, p. 556–560; Tassonyi, Szabó, & Vimláti, 1986, p. 599–616).

In medicine, pipecuronium is used in the form of bromide (Fig. 1, Table 2) for muscle relaxation during

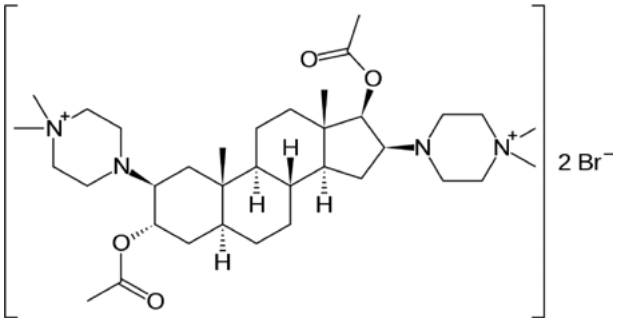

Fig. 1. Structural formula of pipecuronium bromide

Table 2

General characteristics of pipecuronium bromide

(*National Center for Biotechnology Information,* 2021; Rashkovsky, Agoston, & Ket, 1985, p. 1063–1066)

Chemical name	2β, 16β-Bis (4-dimethyl-1-piperazino)-3α, 17β-diacetoxy-5-α-androstane dibromide
Gross formula	$C_{35}H_{62}Br_2N_4O_4$
CAS number	52212-02-9
Molar mass (bases)	762,7 г/моль
Synonyms	Pipicuronium bromide, arduan, aperomide, atracad, vero-pipecuronium, intuban, norcuron, RGH 1106
Visual appearance	The crystalline powder is almost white
Release form	Dry matter in ampoules of white color, transparent colorless solutions (pH = 5,0–6,5)
Solubility	Well soluble in water and ethanol

surgery, including heart surgery and obstetrics and gynecology.

Effective (Karpati, & Biro, 1980, p. 346–354; Aronson, 2016, p. 775–776; Smith, & White, 1993, p. 446–448) pipekuronium bromide dose is 0.03 mg/kg and 0.05 mg/kg. At the same time, a dose of 0.05 mg/kg provides 40–50 minutes of muscle relaxation. The maximum effect is observed in 1.5–5 min, faster if doses of 0.07–0.08 mg/kg are used.

According to the experience of studying potent and poisonous drugs, the physicochemical features of pipecuronium make it impossible to study by thin-layer chromatography, gas chromatography, ultraviolet-visible spectroscopy. Therefore, the author's approbation study used methods of qualitative chemical reactions, mass spectrometry, high performance liquid chromatography with mass spectrometric and corona discharge detection and infrared spectroscopy.

Studies by the method of color tests involve the addition to the aliquot sample of the test substance 2–3 drops of one of the reagents (Table 3), the results are observed for color change.

The results of these qualitative reactions, although not specific for pipecuronium, but may be a part of the analytical scheme of the study.

Due to the fact that pipecuronium is tested mainly in the form of the drug pipecuronium bromide, additional clarification information about it may be the determination of bromide ions (Br-).

To do this, a sample of a specimen weighing 3–4 mg is dissolved in 1 ml of water, taking 3–5 drops of the resulting solution and further diluting in 1 ml of distilled water. To the resulting dilute solution adding 2–3 drops of 5 % aqueous solution of argentum nitrate ($AgNO_3$). In the presence of bromide ions, a pale yellow precipitate is formed, insoluble in nitric acid and easily soluble in sodium thiosulfate solution ($Na_2S_2O_3$) (Studeniak, Voronych, Sukhareva, Fershal, & Bazel, 2014).

Physicochemical characteristics of pipecuronium significantly complicate its study by thin-layer chromatography, the use of silica gel as a stationary phase does not provide positive results, after elution the sample applied to the plate remains at the starting

line. In this case, it may be informative when observing the results of the study (it is not about identification), the nature of the point of application of methanolic solution of pipecuronium after its manifestation. It is applied in the amount of 3–5 μl on a plate for thin-layer chromatography, dried in a stream of warm air and treated, for example, with cobalt rhodonide. After developing the point of application of the pipecuronium solution on the plate, we observe that during the application on the plate the sample of the pipecuronium solution was concentrated docentrically, and the blue spot filled the entire area of the application point from the outer edge to the center (Fig. 2).

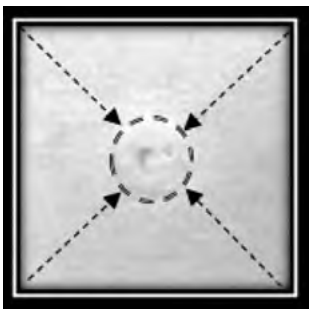

Fig. 2. Image of the manifested point of application of pipecuronium on a plate (Sorbfil) for TLC
Note. The color scheme of the drawing can be observed in the electronic version of the scientific article (https://visnyk.dndekc.mvs.gov.ua/index.php/visnuk)

Among the convenient, from the practice of forensic examination, methods of identification of category A, which are used to obtain information about the molecular structure of the substance – infrared spectroscopy. This is due to the use of modern devices with a diamond crystal (method of disturbed total internal reflection), which allows you to easily analyze most liquid and solid samples without time-consuming sample preparation (native research).

To obtain the spectrum, the test sample (conditions, see Table 4) is pressed against the working surface of the prism (or multiple reflection element), through which the radiation is directed to the spectrophotometer. The sample completely covers the work surface. The spectrum contains bands of two types: characteristic and non-characteristic (Tsomko, Sirenko, & Mazepa, 2012, s. 109–129).

Table 3

Analytical effects of qualitative reactions of a solution of a standard sample of pipecuronium with the most available reagents

Reagent	Color
Marquis	–
Cobalt rhodanide	Blue
Dragendorff	Brown
Wagner	Orange-pink
Potassium iodoplatinate	Dark purple
Nessler	–

Table 4

Conditions of research by IR spectroscopy

Parameter	Terms
Spectrum registration range	4000–525 cm^{-1}
Resolution	4 cm^{-1}
Number of scans	32

The IR absorption spectraum obtained as a result of such a study is a unique physical imprint, a form of reflection of the structure of the test substance, because there are no two compounds (except for optical isomers) that have the same IR spectra for different structures (Kuzmenko, & Churanov, 1977; Lawson, Ogwu, & Tanna, 2018; Naguib, & Abdulatif, 1993, p. 556–560).

Given that today the IR spectrum of pipecuronium is not available in libraries available to expert laboratories, the substance was identified by comparing the vibrational spectrum of the test sample with the spectrum of a standard sample of pipecuronium bromide (Fig. 3) obtained under similar conditions. Characteristic maximum of the absorption bands of the standard sample of pipecuronium are: 3257; 1727; 1452,11; 1248; 1085,15; 1022,66; 931,06 cm^{-1}.

The method of IR spectroscopy, like most spectroscopic methods of analysis, should be used in combination with other methods, so the IR spectrum is used as a source of primary information about the compound of unknown nature.

It is also difficult to objectively identify pipecuronium by the GC-MS method, while the mass

spectral information of the substance can be obtained by the method of direct introduction of the analytical sample into the ionization region (MS-DIP). According to this method, the sample is introduced into the ion source of the device (MS) by means of a valve, rod, conveyor or autosampler; due to the slow evaporation from the micro crucible (DIP), it is possible to study solid and liquid strongly polar, thermolabile and hardly volatile substances or their trace amounts. The method allows to obtain mass spectrum of organic compounds with a relative molecular weight of from 400 to 1000 a. at. m and a boiling point of about 400 °C. Effective mainly for pure substances.

Research by this method, 0.1 μl of the solution or ≈ 1 mg of solid sample is placed in a microtiegel, the analysis is performed under the conditions given in Table 5 (Zamoshets, Barikova, Zelenyi, Kosmina, & Korobchuk, 2021).

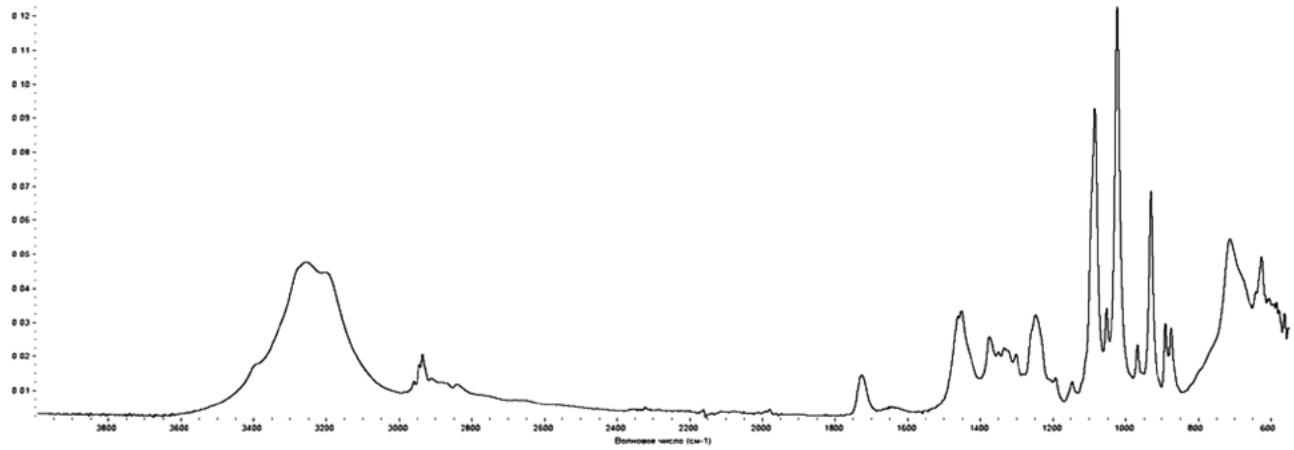

Fig. 3. IR spectrum of a standard sample of pipecuronium bromide

Table 5

Conditions for the study of pipecuronium by MS-DIP

Temperature level	Hold, min	Heating rate, °C/min	Temperature, °C
Start	1	–	40
1	1	0,5	150
2	1	1	250
3 (final)	3	1	390
Total time		10,99	

The study by this method allows to obtain general information about the sample, in case of negative results increase the amount of test substance or change the conditions of the study. The main ions for the identification of pipecuronium by the SIM method: 437; 497; 512 m/z.

A typical chromatogram and mass spectrum of a standard sample of pipecuronium bromide are shown in Fig. 4 and 5.

The most objective method of qualitative research and quantification of pipecuronium is the method of high-performance liquid chromatography based on the mechanisms of adsorption, distribution, ion exchange or size division of molecules.

For such a study, prepare a methanol solution of the test sample with an analyte concentration of 1–1.5 mg/ml, diluted in a ratio of 1:100. Chromatography is performed under the conditions specified in Table 6 (Zamoshets, Barikova, Zelenyi, Kosmina, & Korobchuk, 2021).

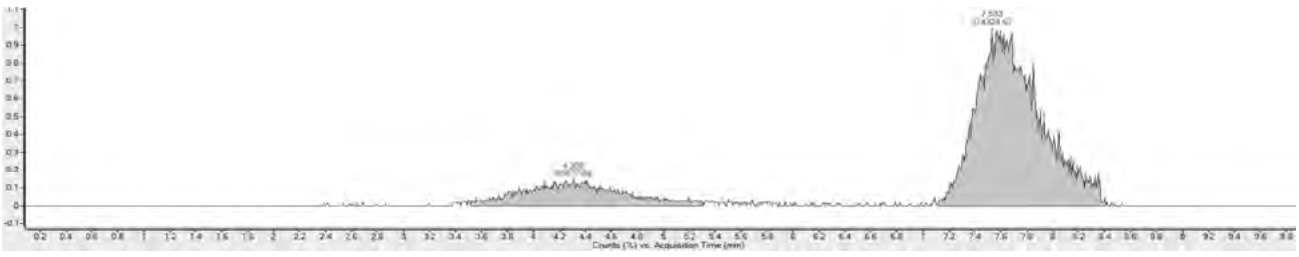

Fig. 4. Typical chromatogram of a standard sample of pipecuronium bromide

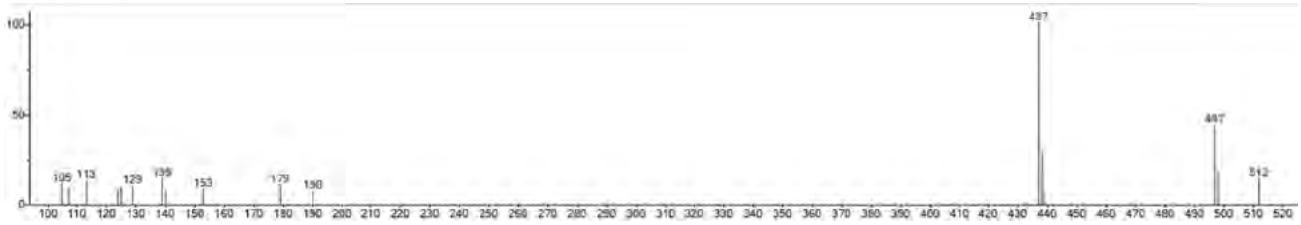

Fig. 5. Mass spectrum of a standard sample of pipecuronium bromide

Table 6

Conditions of chromatography by liquid chromatography

Column	Agilent Poroshel 120 EC C-18, length – 150 mm, diameter – 2,1 mm, phase – 2,7 μm
Moving phase	**A** – 0.1 % aqueous solution of formic acid in water **B** – 0.1 percent methanolic solution of formic acid A:B = 40:60
The speed of the mobile phase	0,400 ml/min
Detector	Mass spectrometric (Q-TOF G6550A) Corona discharge (Thermo Corona Veo RS)
Injection volume	1 mkl

Chromatogram and mass spectrum of a standard sample of pipecuronium bromide, investigated under the conditions specified in Table 6, shown in Fig. 6 and 7.

Pipecuronium, muscle relaxants and steroid compounds using possible variants of other mobile phases (Table 7) were also studied by foreign colleagues (Gazdag, Babják, Kemenes-Bakos, & Görög, 1991, p. 639–644; Błażewicz, Fijałek, & Samsel, 2008, p. 191–195; García, Gomes, Santoro, & Kedor-Hackmann, 2008, p. 1895–1908).

To confirm the results, a parallel study of the samples and checked the convergence of the signals.

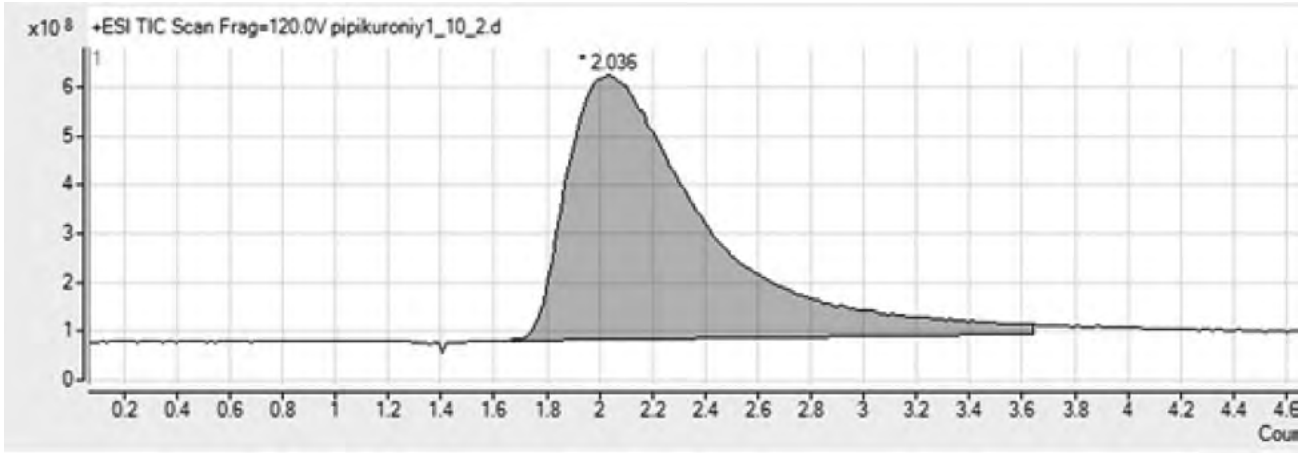

Fig. 6. Typical HPLC chromatogram of a standard sample of pipecuronium bromide obtained under the conditions described in Table 6

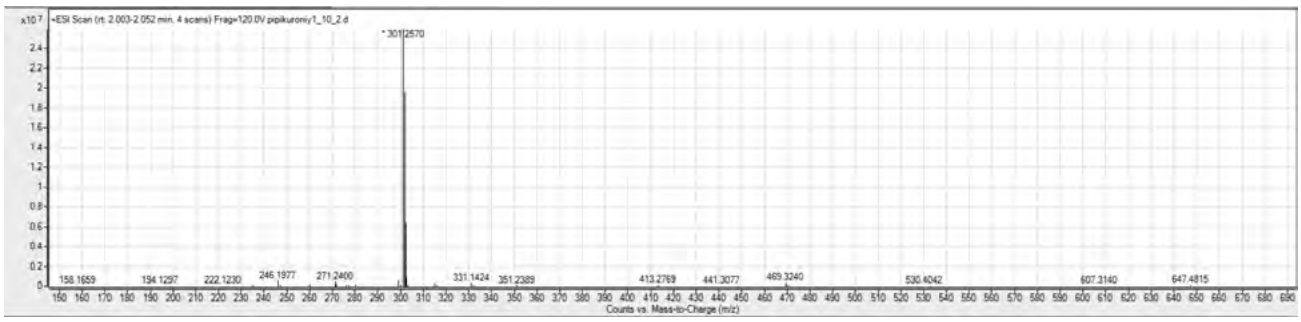

Fig. 7. Mass spectrum of a standard sample of pipecuronium bromide, obtained by HPLC chromatogram

Table 7

Mobile phases used in the study of pipecuronium, muscle relaxants and steroid compounds by HPLC

	Mobile phase 1
Eluent A	4 M $LiClO_4$ – 0,1 M $HClO_4$: water
Eluent B	Acetonitrile
	Mobile phase 2
Eluent A	Water + 0.1 percent formic acid
Eluent B	Acetonitrile / methanol + 0.3 percent formic acid
	Mobile phase 3
Eluent A	100 mm phosphate buffer, pH = 1,8
Eluent B	Acetonitrile

Scientific novelty

The laboratory of the State Scientific Research Forensic Center of the Ministry of Internal Affairs of Ukraine tested the analytical scheme of physicochemical research methods for identification of potent and toxic drugs, recommended by SWGDRUG, and proposed a method of testing a standard sample of pipecuronium by physicochemical methods according to the scheme of analytical methods due to their selectivity.

Conclusions

1. The SWGDRUG recommendations on the combination of analytical methods are analyzed and the minimum requirements for their application are characterized. It should be emphasized that in the context of sufficiency for the identification of potent and toxic drugs, particularly heavy substances, expert laboratories, taking into account the physicochemical properties of such substances and analytical equipment available in expert institutions of the country, must determine a combination of methods to ensure reliable results of analytical research.

2. The possibility of its identification by means of: qualitative chemical reactions (with the most available reagents, such as: Marquis; cobalt rhodonide; Dragendorff; Wagner; potassium iodoplatinate; Nessler), IR spectrometry, mass spectrometry, mass spectrometry liquid chromatography with mass spectrometric and corona discharge detection. The expediency of combining methods is argued, which allows to implement the application of the analytical scheme of research methods recommended by SWGDRUG. It is stated that, given that the use of category B methods is not publicly available, the development of a derivatization process for the study of pipecuronium derivatives by publicly available methods: gas chromatography with mass-selective detection and thin-layer chromatography using different types of sorbents.

3. The results obtained during the approbation of a complex analytical study of pipecuronium according to the recommended international scheme are summarized, and certain proposals are initiated, which can be the basis for research methods of standard sample of pipecuronium by physicochemical methods to confirm the validity, reliability, reproducibility of scientific identification in forensic expert report.

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ІДЕНТИФІКАЦІЯ ПІПЕКУРОНІЮ ЗА СХЕМОЮ ЗАСТОСУВАННЯ АНАЛІТИЧНИХ МЕТОДІВ ДОСЛІДЖЕННЯ ЗАЛЕЖНО ВІД ЇХНЬОЇ СЕЛЕКТИВНОСТІ (SWGDRUG)

Мета статті полягає в комплексному аналізі теоретичних і практичних аспектів ідентифікації піпекуронію відповідно до схеми застосування аналітичних методів дослідження, зумовленої їхньою селективністю, та розробленні методики дослідження стандартного зразка піпекуронію фізико-хімічними методами для підтвердження аргументованості, достовірності, відтворюваності результатів його ідентифікації та достатності для підготовки науково обґрунтованого висновку судового експерта. ***Методологія***. Достовірність отриманих результатів і висновків забезпечено використанням комплексу загальнонаукових і спеціальних методів дослідження. Методи аналізу, синтезу, порівняння, узагальнення дозволили проаналізувати інформаційні джерела за напрямом дослідження, а також аналітичну схему комплексу фізико-хімічних методів дослідження, рекомендовану SWGDRUG. Апробаційне аналітичне дослідження стандартного зразка піпекуронію із застосуванням методів експерименту, аналізу, порівняння, а також спеціальних фізичних, хімічних, статистичних методів дослідження дозволило апробувати комплекс фізико-хімічних методів дослідження цієї речовини й дійти висновків щодо достатності для цілей дослідження певних їх видів та окреслити напрями подальших науково-дослідних розвідок. ***Наукова новизна***. В умовах лабораторії Державного науково-дослідного експертно-криміналістичного центру МВС України апробовано аналітичну схему фізико-хімічних методів дослідження для ідентифікації сильнодійних та отруйних лікарських засобів, рекомендовану SWGDRUG, і запропоновано методику дослідження стандартного зразка піпекуронію фізико-хімічними методами відповідно до схеми застосування аналітичних методів дослідження, зумовленої їхньою селективністю. ***Висновки***. Проаналізовано рекомендації SWGDRUG стосовно комбінації аналітичних методів і схарактеризувано мінімальні вимоги для їх застосування. При цьому наголошено, що в контексті достатності для ідентифікації сильнодійних і отруйних лікарських засобів, зокрема важколетких речовин, експертні лабораторії, зважаючи на фізико-хімічні властивості таких речовин і наявне в експертних установах країни аналітичне обладнання, мають самостійно визначати комбінацію методів, щоб забезпечити достовірність результатів аналітичних досліджень. Засвідчено комплексним аналітичним дослідженням стандартного зразка піпекуронію можливість його ідентифікації за допомогою методів: якісних хімічних реакцій (із найбільш доступними реактивами, такими як: Маркі, родонід кобальту, Драгендорфа, Вагнера, йодоплатинат калію, Несслера), ІЧ-спектрометрії, мас-спектрометрії, високоефективної рідинної хроматографії з мас-спектрометричним та коронорозрядним детектуванням. Аргументовано доцільність комбінування методів, що дозволяє реалізовувати застосування аналітичної схеми методів дослідження, рекомендованої SWGDRUG. Констатовано необхідність, з огляду на те, що використання методів категорії В не є загальнодоступним, розроблення процесу дериватизації для дослідження похідних піпекуронію загальнодоступними методами: газовою хроматографією з мас-селективним детектуванням і тонкошаровою хроматографією з використанням різних видів сорбентів. Узагальнено результати, отримані під час апробації комплексного аналітичного дослідження піпекуронію за рекомендованою міжнародною схемою, і започатковано певні пропозиції, які можуть стати підґрунтям методики дослідження стандартного зразка піпекуронію фізико-хімічними методами для підтвердження аргументованості, достовірності, відтворюваності результатів його ідентифікації та достатності для підготовки науково обґрунтованого висновку судового експерта.

Ключові слова: судова експертиза; судовий експерт; висновок судового експерта; SWGDRUG; аналітичні методи дослідження, використовувані SWGDRUG; селективність; аналітична схема методів дослідження; ідентифікація сильнодійних і отруйних лікарських засобів; важколеткі речовини; піпекуронію бромід.

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ИДЕНТИФИКАЦИЯ ПИПЕКУРОНИЯ СОГЛАСНО СХЕМЕ ПРИМЕНЕНИЯ АНАЛИТИЧЕСКИХ МЕТОДОВ ИССЛЕДОВАНИЯ, ОБУСЛОВЛЕННОЙ ИХ СЕЛЕКТИВНОСТЬЮ (SWGDRUG)

Цель статьи заключается в комплексном анализе теоретических и практических аспектов идентификации пипекурония согласно схеме применения аналитических методов исследования, обусловленной их селективностью и разработке методики исследования стандартного образца пипекурония физико-химическими методами для подтверждения аргументированности, достоверности, воспроизводимости результатов его идентификации и достаточности для подготовки научно обоснованного заключения судебного эксперта. ***Методология***. Достоверность полученных результатов и выводов обеспечена использованием комплекса общенаучных и специальных методов исследования. Методы анализа, синтеза, сравнения, обобщения позволили проанализировать информационные источники по направлению исследования, а также аналитическую схему комплекса физико-химических методов исследования, рекомендованную SWGDRUG. Апробационное аналитическое исследование стандартного образца пипекурония с применением методов эксперимента, анализа, сравнения, а также специальных физических, химических, статистических методов исследования позволило апробировать комплекс физико-химических методов исследования этого вещества и сделать выводы относительно достаточности для целей исследования определенных их видов и наметить направления дальнейших научно-исследовательских изысканий. ***Научная новизна***. В условиях лаборатории Государственного научно-исследовательского экспертно-криминалистического центра МВД Украины апробирована аналитическая схема физико-химических методов исследования для идентификации сильнодействующих и ядовитых лекарственных средств, рекомендованная SWGDRUG, и предложена методика исследования стандартного образца пипекурония физико-химическими методами в соответствии со схемой применения аналитических методов исследования, обусловленной их селективностью. ***Выводы***. Проанализированы рекомендации SWGDRUG относительно комбинации аналитических методов и описаны минимальные требования для их применения. При этом отмечено, что в контексте достаточности для идентификации сильнодействующих и ядовитых лекарственных средств, в частности труднолетучих веществ, экспертные лаборатории, учитывая физико-химические свойства таких веществ и имеющееся в экспертных учреждениях страны аналитическое оборудование, должны самостоятельно определять комбинацию методов, чтобы обеспечить достоверность результатов аналитических исследований. Комплексным аналитическим исследованиям стандартного образца пипекурония подтверждена возможность его идентификации с помощью методов: качественных химических реакций (с наиболее доступными реактивами, такими как: Марки, родонид кобальта, Драгендорфа, Вагнера, йодоплатинат калия, Несслера), ИК-спектрометрии, масс-спектрометрии, высокоэффективной жидкостной хроматографии с масс-спектрометрическим и коронорозрядним детектированием. Аргументирована целесообразность комбинирования методов, что позволяет реализовывать применение аналитической схемы методов исследования, рекомендованной SWGDRUG. Констатирована необходимость, учитывая то, что использование методов категории В не является общедоступным, разработки процесса дериватизации для исследования производных пипекурония общедоступными методами: газовой хроматографии с масс-селективным детектированием и тонкослойной хроматографии с использованием различных видов сорбентов. Обобщены результаты, полученные при апробации комплексного аналитического исследования пипекурония по рекомендованной международной схеме, и сделаны некоторые предложения, которые могут стать основой методики исследования стандартного образца пипекурония физико-химическими методами для подтверждения аргументированности, достоверности, воспроизводимости результатов

его идентификации и достаточности для подготовки научно обоснованного заключения судебного эксперта.

Ключевые слова: судебная экспертиза; судебный эксперт; вывод судебного эксперта; SWGDRUG; аналитические методы исследования, используемые SWGDRUG; селективность; аналитическая схема методов исследования; идентификация сильнодействующих и ядовитых лекарственных средств; труднолетучие вещества; пипекурония бромид.