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DEVELOPMENT OF A METHODOLOGY FOR EXPERT RESEARCH OF SMALL AMOUNTS OF MEFEDRON USING GAS CHROMATOGRAPHY METHODS WITH A MASS SPECTROMETRIC DETECTOR AND IR SPECTROPHOTOMETRY

Açar sözlər: nəzarət olunan maddə, narkotik vasitələr, kütlə-spektrometrik detektorlu qaz xromatoqrafiyası (GC-MS)

Key words: controlled substance, narcotic drugs, gas chromatography with mass spectrometric detector (GC-MS)

Anahtar kelimeler: kontrollü madde, narkotik ilaçlar, kütle spektrometrik dedektörlü gaz kromatografisi (GC-MS)

Ключевые слова: контролируемое вещество, наркотические средства, газовая хроматография с масс-спектрометрическим детектированием (ГХ-МС)

The problem of drug addiction is one of the most important international problems of today. Combating drug smuggling is one of the main tasks of law enforcement agencies in all states. New types of drugs, especially synthetic ones, are becoming increasingly widespread throughout the world today. Although full information about causing grievous bodily and psychological harm is provided in the mass media, the fight against this evil is not sufficiently effective, which is reflected in forensic practice.

Expert examination of narcotic drugs, in particular those obtained by synthetic means, often involves not only determining their

chemical nature, properties, and structure, but also one of the most complex tasks - identifying their minimal concentrations in various objects. For solving such problems, the mass spectrometric detector gas chromatography method has high selectivity, allowing in a number of cases to obtain the necessary information about the composition and structure of the studied compounds. Analysis of expert practice on the use of gas chromatography with a mass spectrometric detector in the analysis of narcotic drugs demonstrates its wide possibilities in cases where the informativeness of other analytical



methods is insufficient [1, P.17-18].

The mass spectrometric detector gas chromatography method is the most optimal option when it is possible to obtain maximum information about the substance (retention time, molecular and fragmentation ions, peak intensity) in the presence of trace amounts. At the same time, the speed, relative simplicity of sample preparation, accuracy, and reproducibility of the obtained results are undeniable.

IR spectrophotometry is also used in various fields of science, including forensic practice. Using this analysis method, it is possible to identify organic substances in various aggregate states (gas, liquid, solid). IR spectrophotometry determines the inertia of a substance in a molecule per unit time. Knowing the mass of a substance's atoms allows us to determine the distance between atoms and the angles of their interaction.

To determine the IR spectrum of an unknown substance, IR spectroscopes are used. Based on the results of the obtained spectrum, the types of vibrations and bonds of the functional groups of the substance molecule, as well as the structure of the substance, are determined [2, p. 3].

The Republican Center for Forensic Examination named after X. Sulaymanova was presented by the judicial authorities with a material evidence - a plastic bag containing cream-colored powder residues, seized from the location of the corpse of citizen S. The experts were asked questions: Are there any residues of narcotic drugs in the plastic bag, and if so, to which group do they belong?

To accomplish this task, a spirit wash was prepared from the inner surface of the polyethylene bag. For this, 1 ml of 96% ethyl alcohol was poured into a bag and extraction was carried out in a "SONOREX" brand ultrasonic bath at a temperature of 40° C for 10 minutes. The obtained wash was evaporated at room temperature to a volume of 100 pl and used for further analysis.

Chromato-mass spectrometric studies were conducted on an AT 5973 chromato-mass spectrometer using the Drug SP-SHORTSPLIT-LESS-100H2.M method (HP5MS capillary column, 30 m long, 0.25 mm diameter, with 5% phenylmethylsiloxane, mass- selective de-

tector) under the following analysis conditions: ionizing electron energy of 70 eV, injector temperature of 280°C, furnace temperature from 150°C to 280°C in programmed mode with a temperature rise rate of 15°C per minute, sample value of 1 pl, test substance vapor pressure of 10 mm Hg, analysis time of 20 min, gas-carrier-hydrogen, flow rate of 2.1 ml/min, flow division mode of 10:1.

Analysis of the obtained chromatogram and mass spectrum indicates that the mass spectrum of the investigated washing is characterized by the presence of stable fragments, characteristic ions formed along the general paths of molecular ion fragmentation. Figures 1 and 2 show the chromatogram and mass spectrum of washing from the packet. The chromatogram and mass spectrum of the extract were identified using a database library called NIST0 2.L., NIST 11.L., Wiley225.L, SWDRUG.L., CAYMAN-SPECTRA.L., SWDRUG3.5.L. [4].

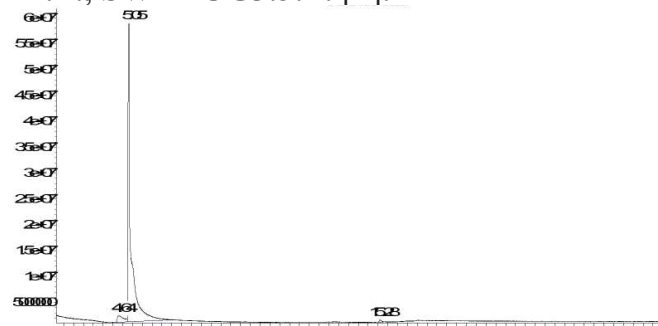
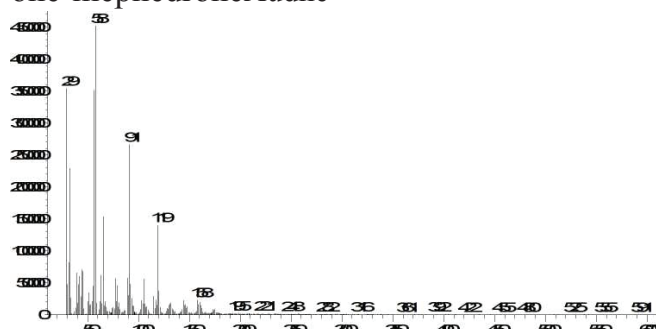


Fig. 1. Chromatogram of washing from the test packet

Thus, in the chromatogram of washing from the packet, the main peak with a retention time of 5.00 min. was detected, and its mass spectrum is characterized by fragmentation ions m/z 177, 147, 119, 91, 58. Based on the results of studying the obtained chromatogram and mass spectrum and comparing them with the database, it was established that the identified chromatographic and mass spectrometric parameters correspond to 4-methylmethionone-mephedrone.





Andes

Fig. 2. Washing mass spectrum from the test packet

The wash residue from the packet was evaporated to dryness, and IR spectral analysis was performed on an AGILENT TECHNOLOGY FTIR-640 IR spectrometer using the NCPV (violated full internal reflection) attachment under the following analysis conditions: registration range 2954-422 cm⁻¹, number of scanners -12. The identification of IR spectra was carried out based on comparing the obtained IR spectra with standard spectra from the IR spectrum library database available in the device.

In this case, an IR spectrum with characteristic bands of 2905, 2796, 2713, 1685, 1605, 1455, 1434, 1357, 1296, 1205, 1186, 968, 898, 755 cm⁻¹ was obtained, which are due to valence and deformation vibrations of free and bound methyl (-CH₃), methylene (-CH₂-) groups, free and bound amino (-NH₂-) and carbonyl (-C=O) groups. The functional groups identified by the characteristic bands of transmission are characteristic of the structure of 4-methylmethcathinone- mephedrone, which also coincides with the data available in the device's library database (see Fig. 3).

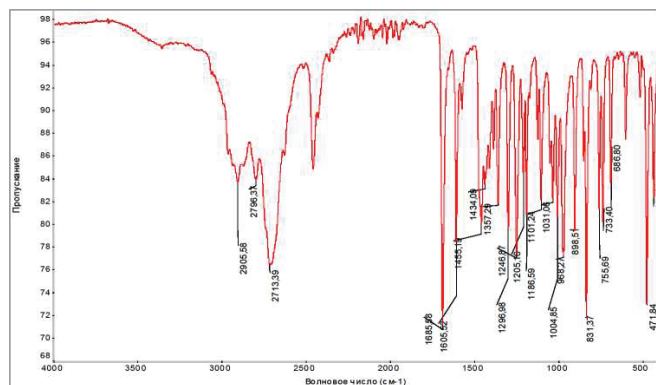
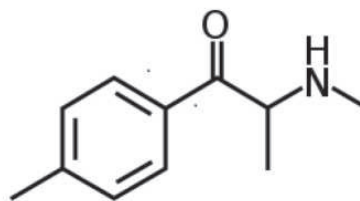


Fig. 3. IR spectrum of washing from the test packet

Thus, as a result of research using the gas chromatography method with a mass spectrometric leak detector from the test package, the presence of 4-methylmethcathinone- mephedrone was established. The molecular ion (177) and fragmentation ions (m/z 177, 147, 119, 91, 58) obtained in the spectrum, as well as the IR spectrum of washing with functional groups identified along the characteristic bands of conduction, characteristic of the 4-methyl methotone-me-

phedrone structure, confirm its molecular weight and structural formula, which has the following structure:

Mephedrone



Systematic name: (RS) -2-methylamino-1- (4-methylphenyl) propane-1-one

Traditional name: mephedrone, 4-methyl methathionine C₁₁H₁₅NO

White easily breakable crystals

177.2429 g/mol

Mephedrone, also known as 4-methylmethcathinone (shortly 4-MMC, from English 4- methylmethcathinone) or 4-methylephedrone, is a chemical compound of the class of substituted amphetamines and cathinones, a psychostimulant and empathogen.

English jargon names: mephstasy (mephstasy), meow meow (meow-meow), (due to the abbreviation "4-MMCathinone" ^ "meow meow cat"), Cristalius (crystallium) [5], cat piss (cat's urine), mad hen (mad hen), mad cow (mad cow), bubble/bubbles (bubble, bubbles), M-Cat [6], TopCat [7]. In the USA and Europe, mephedrone was sold for some time as bath salt [8, P.399], food additive, plant fertilizer, and rodent remedy.

In Russia and the countries of the post-Soviet space, the most commonly used jargon names are: salt, meph, miau, grandfather, crystals, met. [9].

The following negative effects are observed when taking mephedrone:

rapid heartbeat (tachycardia); short-term memory impairment;

insomnia; jaw muscle spasm; muscle twitching; eccentric behavior; decreased concentration; nystagmus; dizziness; sometimes severe narrowing of blood vessel and artery lumens; vasoconstriction);

When taken intranasally: burning in the nose and pharynx, irritation of the mucous membrane, ulcers form at the points of contact with the substance.

List of main side effects based on EMCDDA data [10], which represent typical symptoms of



stimulatory psychosis: strong desire to repeat drug use (withdrawal syndrome); depression; pursuit delusion; hallucinations, delirium, delirium; panic attacks; insomnia; loss of appetite (anorexia); increased heart rate (tachycardia); elevated blood pressure; uncomfortable changes in body temperature (fever or chills); dizziness; muscle aches and weakness, seizures, tremors; numbness of limbs; visual snow.

Acute mephedrone intoxication causes euphoria, excitement, perception of various music, improves mood, reduces hostility, increases sociability, accelerates the associative process, increases sexual arousal [11, P. 399]. From the physical negative manifestations, one can note the difficulty in achieving ejaculation and the blue color of the lips and extremities (cyanosis), and both problems are associated with vasoconstriction. Such effects of mephedrone are similar to the effects of cocaine, amphetamines, and MDMA. Of the 70 Dutch people who used mephedrone, 58 people called mephedrone use a pleasant experience, and 12 - an unpleasant one [12]. In a survey of British mephedrone users who had previously used cocaine, most found that the effects were longer-lasting but less captivating. Users were also asked to compare the “risks” of mephedrone and cocaine use, and they responded that the “risk” from mephedrone was higher [13]. A study of mephedrone users from Northern Ireland concluded that most of them considered mephedrone dangerous to use.

In the Republic of Uzbekistan, Mephedrone, according to the Resolution of the Cabinet of

Ministers of the Republic of Uzbekistan No. 330 dated November 12, 2015, “On the Import, Export, and Transit of Narcotic Drugs, Psychotropic Substances, and Precursors through the Territory of the Republic of Uzbekistan,” is included in the list of narcotic drugs prohibited for use in the territory of the Republic of Uzbekistan (List I, position 97) [14].

Based on the analysis results using mass spectrometric detector gas chromatography and IR spectrophotometry methods, the following were established: the presence of mephedrone traces in the investigated polyethylene packet; retention time, molecular and fragmentation ions, their intensity, and the individuality of mephedrone molecule fragmentation. IR spectrum with characteristic bands of functional groups characteristic of the mephedrone structure. These parameters are recommended for analyzing small quantities of unknown substances, as well as for determining the genus and group affiliation and the common source of their origin.

Thus, a method for forensic examination of small amounts of mephedrone in a polyethylene bag using gas chromatography with a mass spectrometric detector and IR spectrophotometry has been developed. It has been proven that the application of these methods, which possess high sensitivity, speed, and ease of use, allows for the rapid and high-precision (10-12g) identification of unknown substances in the composition of objects submitted for expert examination.

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S U M M A R Y

A technique for expert study of small quantities of mephedrone on carrier objects has been developed using the method of gas chromatography with a mass spectrometric detector and IR spectrophotometry.

The retention time molecular and fragment ions, their intensity, individuality, fragmentation of substance, as well as the presence of characteristic transmission bands of functional groups characteristic of the structure of this compound.

It has been established that the application of this method, which has a high sensitivity, speed and ease of use, makes it possible to quickly and with high accuracy identify unknown substances in the composition of micro-objects entering for expert research.

These methods have been proven to be highly sensitive, fast and easy to use.

KÜTLƏ SPEKTROMETRİYA DETEKTORU VƏ İNFRAQIRMIZI SPEKTROFOTOMETRİYA İLƏ QAZ XROMATOQRAFIYASI METODLARINDAN İSTİFADƏ EDƏRƏK AZ MİQDARDA MEFEDRON ÜZƏRİNDƏ EKSPERT TƏDQİQATI METODOLOGİYASININ HAZIRLANMASI

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X Ü L A S Ə

Daşıyıcı obyektlərdə mefedronun kiçik miqdarının ekspert tədqiqi üçün bir texnika kütlə spektrometrik detektoru və İQ spektrofotometriyası ilə qaz xromatografiyası metodundan istifadə etməklə hazırlanmışdır.

Molekulyar və fraqment ionlarının saxlanma müddəti, onların intensivliyi, fərdiliyi, maddənin fraqmentasiyası, eləcə də bu birləşmənin strukturuna xas olan funksional qrupların xarakterik ötürmə zolaqlarının mövcudluğu.

Yüksək həssaslığa, sürətə və istifadə rahatlığına malik olan bu metodun tətbiqinin ekspert tədqiqatı üçün daxil olan mikroobyektlərin tərkibindəki naməlum maddələri tez və yüksək dəqiqliklə müəyyən etməyə imkan verdiyi müəyyən edilmişdir.

Bu metodların yüksək həssaslıq, sürətli və istifadəsi asan olduğu sübut edilmişdir.

KÜTLE SPEKTROMETRİ DEDEKTÖRÜ VE IR SPEKTROFOTOMETRİ İLE GAZ KROMATOĞRAFİSİ YÖNTEMLERİ KULLANILARAK AZ MİKTARDA MEFEDRONUN UZMAN ARAŞTIRMASI İÇİN BİR METODOLOJİNİN GELİŞTİRİLMESİ

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Ö Z E T

Taşıyıcı nesneler üzerindeki küçük miktarlardaki mefedronun uzman incelemesi için, kütlə spektrometrik dedektörlü gaz kromatografisi və IR spektrofotometresi yöntemi kullanılarak bir teknik geliştirilmiştir.

Moleküler ve parçacık iyonlarının tutunma süreleri, yoğunlukları, bireysellikleri, maddenin parçalanması ve bu bileşiğin yapısına özgü fonksiyonel grupların karakteristik iletim bantlarının varlığı incelenmiştir.

Yüksek hassasiyet, hız ve kullanım kolaylığına sahip bu yöntemin uygulanmasının, uzman araştırmasına giren mikro nesnelerin bileşimindeki bilinmeyen maddeleri hızlı ve yüksek doğrulukla tanımlamayı mümkün kıldığı tespit edilmiştir.

Bu yöntemlerin son derece hassas, hızlı ve kullanımı kolay olduğu kanıtlanmıştır.



РАЗРАБОТКА МЕТОДОЛОГИИ ДЛЯ ЭКСПЕРТНОГО ИССЛЕДОВАНИЯ МАЛЫХ КОЛИЧЕСТВ МЕФЕДРОНА С ИСПОЛЬЗО- ВАНИЕМ МЕТОДОВ ГАЗОВОЙ ХРОМАТОГРАФИИ С МАСС-СПЕКТРОМЕТРИЧЕСКИМ ДЕТЕКТОРОМ И ИК-СПЕКТРОФОТОМЕТРИЕЙ

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Р Е З Ю М Е

Разработана методика экспертного исследования малых количеств мефедрона на объектах-носителях с использованием метода газовой хроматографии с масс-спектрометрическим детектором и ИК-спектрофотометрии.

Определяются время удерживания молекулярных и фрагментных ионов, их интенсивность, индивидуальность, фрагментация вещества, а также наличие характерных полос пропускания функциональных групп, характерных для структуры данного соединения.

Установлено, что применение данной методики, обладающей высокой чувствительностью, скоростью и простотой использования, позволяет быстро и с высокой точностью идентифицировать неизвестные вещества в составе микрообъектов, поступающих на экспертизу.

Эти методы доказали свою высокую чувствительность, быстроту и простоту использования.

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