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METHODOLOGY FOR STUDYING TRACE QUANTITIES OF UNKNOWN SUBSTANCE USING GC-MS

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Abstrakt: *The article presents the methodology of forensic chemical research of trace amounts of unknown substances using gas chromatography-mass spectrometric analysis.*
Established: retention time, molecular and fragmentation ions, their intensity, individual fragmentation of a substance, name, chemical structure and structural freemule.
These parameters are recommended for the detection of controlled substances in complex matrices.
This method has proven to be very sensitive, fast and easy to use.
Key words: *controlled substances, narcotic drugs, local anesthetic, gas chromatography-mass spectrometry, retention time, molecular and fragmentation ions.*

Introduction. In the investigation and trial of criminal cases related to drug trafficking, micro-objects found at the crime scene, as well as on objects related to the crime event and its participants, acquire an increasing evidentiary value.

Forensic chemical research of micro-quantities of narcotic drugs in relation to 0.1 - 0.5 mg of a substance has a number of distinctive features associated with the improvement of the methods used, which is due to the small volume of substances, the multiplicity of their forms, a variety of objects - carriers, the specifics of the relationship of micro-quantities of substances with objects - carriers, etc. Trace amounts of narcotic drugs are often used in investigative, judicial and expert practice as a source of information about the circumstances of a criminal case, since information about the nature of the drug, the place of discovery of the micro-object, the type of object-carrier is valuable for solving many issues. It allows you to determine the location and method of manufacture of drugs, as well as the source of their purchase and sale. In expert practice, the objects of research are often narcotic drugs, which are rarely individual compounds. At the same time, the greatest difficulties arise in the study of substances that are multicomponent systems and mixtures containing a large amount of impurity and ballast substances, as well as low concentrations of active substances. Difficulties arising in the study of such objects are due to the following reasons:

-Complexity of separation of multicomponent systems and cleaning from ballast substances and impurities;
-Complexity of concentration of dilute solutions.

All this complicates the identification of the substance and served as the basis for the development of a methodology for the study of trace amounts of narcotic drugs.

The trend in the development of forensic examination shows that an increasing number of research methods from various fields of science and technology are used in it, the technical equipment of expert institutions and research methods in the production of examinations are being improved more and more [1].

In the field of forensic chemical research of a micro-object of narcotic drugs, the key to obtaining new information is the use of more adequate and accurate research methods and techniques. Despite the fact that gas chromatographic analysis is the most common method for analyzing drugs, recently in the world practice of forensic chemical and medical expertise, gas-liquid chromatography with a mass spectrometric detector (GC-MS), which makes it possible to identify all volatile substances ...

The proposed technique was tested in the study of drugs such as heroin, morphine, opium, fentanyl, etonitazene, methamphetamine, etc.

General questions of the study of micro-quantities of narcotic drugs. Due to the fact that objects entering forensic chemical research exist in extremely diverse forms (free-flowing, viscous, liquid, compressed, resinous, granular, etc.), it is very difficult to define quantitative criteria for a micro-object. From expert practice, we noted that compliance with any specific requirements of objects submitted for expert examination in terms of the size of individual particles, mass, or volume is not always observed and is not always possible, since often material evidence is received for research with barely noticeable visual inspection with traces of foreign substances on their surface. And sometimes, the presence of drugs is only assumed by the investigator within the circumstances of the case. Thus, micro-amounts of narcotic drugs are often present on media objects in the form of layers, overlays, single particles, etc.

It has been experimentally established that the minimum amount of a narcotic drug that allows a study to be carried out in the form of a solid sample is an amount of 1 mg, in the form of a liquid - 1 mL. However, in the case of the study of trace amounts of these substances on various media objects (tissue, paper, washes from hands, subungual contents, syringes, needles, etc.), these quantification data are not applicable [2].

When examining micro-quantities of narcotic drugs on various carrier objects, the expert is asked questions concerning:

- establishing the nature of objects and classifying them as narcotic drugs;
- establishing a common group affiliation based on a single source of origin, etc.

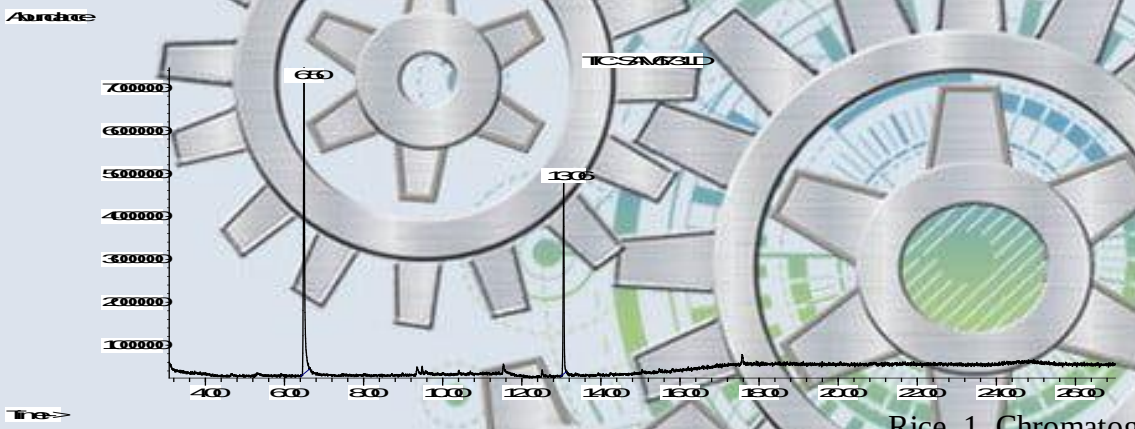
The aim of the study is to use the method of chromatography-mass spectrometry in the forensic chemical analysis of trace amounts of unknown drugs on carrier objects. So, in the laboratory of forensic research of materials, substances and products of the Republican Center for Forensic Examination named after Kh.Sulaimanova from the judicial and investigative authorities received material evidence, seized from the place of discovery of the corpse of gr. V. Arkhipov. Along with other physical evidence, the study received a cardboard lid from a shoe box. The lid on the inside has dirt and a white spot. The experts were asked what traces of a substance are on the inner surface of the box lid and whether this substance is included in the list of narcotic or psychotropic substances. The initial stage in the study of an object received for examination - a lid in order to detect traces of poisonous, potent, narcotic drugs and psychotropic substances on it, is the extraction of a possibly present substance from the contaminated parts of the lid.

For this, its inner surface was thoroughly wiped with a swab moistened with ethyl alcohol, then a part of the lid with white spots was cut out, crushed, and poured together with a swab, which was used to wipe the inner surface of the lid , with a minimum amount of 96% ethyl alcohol, ensuring the sample coverage. The extraction was carried out for 6 hours, then the alcoholic extract was decanted, evaporated to a volume of 100 microL and used for further analysis. The second stage in the study of trace amounts of poisonous, potent, narcotic drugs is the detection of active components and related substances by instrumental methods [3,4].

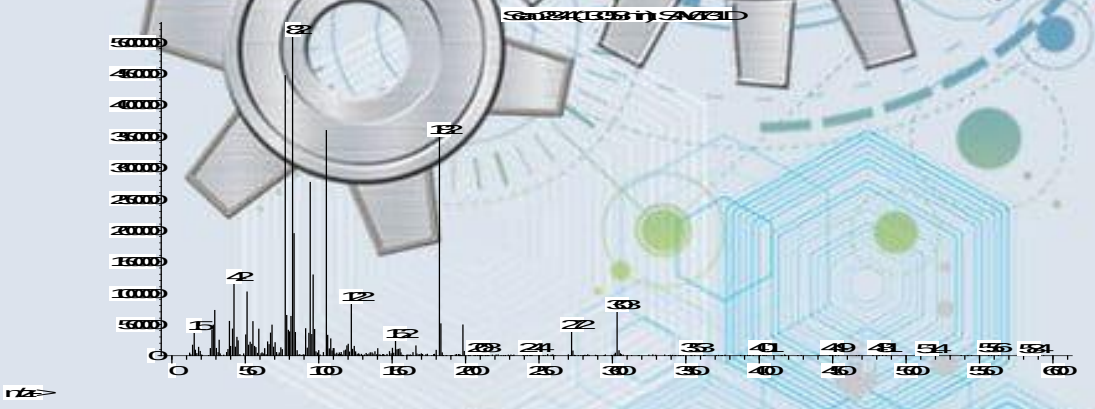
Materials and research methods

Chromato-mass spectrometric study was carried out on an AT 5973 gas chromatograph-mass spectrometer by the Drug SP-SHORTSPLITLESS-100H2.M method (capillary column HP5MS, 30 m long, 0.25 mm in diameter, with 5% phenylmethylsiloxane, mass-selective detector) under the following analysis conditions: ionizing electron energy 70 eV, injector temperature 280 ° C, oven temperature from 150 ° to 280 ° C in programmed mode with a temperature rise rate of 15 ° C per minute, sample size 1micro mL, vapor pressure of the test substance 10 mmHg Art., analysis time - 20 min, carrier gas - hydrogen, flow rate - 2.1 ml / min, in a mode with a flow division of 10: 1.

Results and its discussion. Analysis of the obtained chromatograms and mass spectra indicates that the mass spectra of the investigated extract are characterized by the presence of stable fragments, characteristic ions formed along the common pathways of fragmentation of molecular ions. Below is their chromatogram and mass spectrum (Figures 1, 2). Chromatogram and mass spectrum of the swab extract and crushed parts of the box lid are identified using a database library named NIST02.L., NIST11.L., Wiley225.L., SWDRUG.L., CAYMAN-SPECTRA.L., SWDRUG3 .5.L. [5, 6].



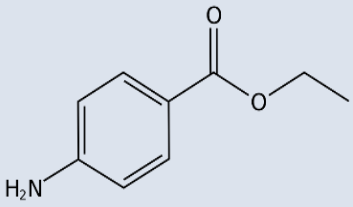
Rice. 1. Chromatogram of the extract from the swab and crushed parts box lids



Rice. 2. Mass spectrum of swab extract and crushed parts box lids

It was found that the main peak, with a retention time of 6.50 min. and fragmentation ions with m / z 165, 120, 92, 65 corresponds to benzocaine, and the second peak with retention time 13.06 min. and fragmentation ions with m / z 303, 182, 82, 105, 42 corresponds to cocaine.

Benzocaine

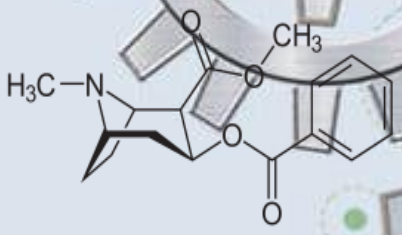


Chemical Name:
4-Aminobenzoic acid ethylester
Gross formula $C_9H_{11}NO_2$
Molecular weight 165.189

Benzocaine ("Anestezin") is a drug, local anesthetic.
Applied: inside for gastralgia, esophagitis, gastric ulcer and duodenal ulcer.
Locally: acute inflammation of the middle ear, pain in the area of the external auditory canal, urticaria, skin diseases accompanied by itching; perianal fissures, hemorrhoids. Performing diagnostic manipulations on mucous membranes

(gastroscopy, rectoscopy, otoscopy, ureteroscopy, gynecological procedures). In dentistry: surface anesthesia.

Cocaine



Chemical name: Methyl (1R12R13S15S) - 3 benzoyloxy) – 8-methyl-8- azabicyclo [3.2.1] – octane-2- carboxylateMethyl (1R12R13S15S) - 3 (benzoyloxy) – 8-methyl-8- azabicyclo [3.2.1] – octane-2- carboxylate

Traditional name: cocaine, ecgonyl benzoate, L-cocaine, (R) - (-) - cocaine
Gross formula: base C17H21NO4, hydrochloride: C17H22NO4Cl
Molecular weight: 303, 35.

Cocaine is a tropane alkaloid, a methyl ester of benzoyl ecgonyne, and a widespread drug. It has a local anesthetic effect and a powerful stimulating effect on the central nervous system of a person, causing a feeling of euphoria.Initially, it was widely used for medical purposes, but by the beginning of the 20th century it was almost completely ousted from medical practice by more advanced drugs. Currently, it is the second most important "problem drug" after opiates - a narcotic substance, the abuse of which is a significant socio-economic problem. Regular use of cocaine is psychologically addictive. With long-term use of cocaine, negative clinical effects appear, including disturbances in sleep, memory and attention, fatigue, weight loss, arrhythmia, angina pectoris, depression and suicidal tendencies, obsessions and hallucinations; cerebral stroke and myocardial infarction are possible.

Thus, as a result of gas chromatography-mass spectrometric study flush obtained from the carrier object - the lid from the box, the presence of traces of benzocaine and cocaine. Benzocaine is a local anesthetic drug and is not included in the list of narcotic drugs and psychoactive substances. Cocaine, 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, the circulation of which is limited in the territory of the Republic of Uzbekistan. (List II, 15-position) [7].

Conclusions. According to the results of the analysis by gas-liquid chromatography with a mass spectrometric detector, it was established: the presence on the inner surface of the investigated cover from the box of benzocaine and cocaine; retention time, molecular and fragment ions, their intensity, individual fragmentation of benzocaine and cocaine molecules.

For the analysis of trace amounts of unknown substances on objects of carriers, as well as for establishing the generic and group affiliation and the common source of their origin, it is recommended to use these parameters.

Thus, a technique has been developed for the forensic chemical study of trace amounts of cocaine in a mixture on a carrier object using the method of gas-liquid chromatography with a mass spectrometric detector. It has been proven that the use of this method, which has high sensitivity, speed and ease of use, makes it possible to quickly and with high accuracy (about 10-12 g) identify unknown substances in the composition of mixtures at objects submitted for expert research.

References:

1. Islamov T.Kh., Bakanova L.P. Expansion of opportunities for the study of micro-objects in the investigation and disclosure of crimes, Academy of the Ministry of Internal Affairs of the Republic of Uzbekistan, Tashkent, 2009, 8 p.;

2. Islamov T.Kh., Bakanova L.P. Features of forensic examination of micro-quantities of narcotic and medicinal products, Academy of the Ministry of Internal Affairs of the Republic of Uzbekistan, Tashkent, 2009, 9 p.;

3. Sidametova Z. E., Olimov N. K. Quality Assessment of Sedative Medicinal Plants and Their Remedies. RA journal of applied research.-2022.- Vol.08.-Issue 03.- P. 197-203. (SJIF =7,036).

4. Sidametova Z. E., Olimov N. K. Quality Assessment of Active Substances that Demonstrate Sedative Effect of «Flegmen» Syrup. Global Journal of Medical Research.-2022.- Vol. 22.-Issue 2.- P. 43-48. (SJIF =8,23).

5. K. Pfleger, H. Mauere, A. Weber. Mass Spektral and GS Data of Drugs. Part 2, 3. New York. 2010.

6. Clarkes “Isolation and identification of drugs”. London, 2016.

7. Resolution of the Cabinet of Ministers of the Republic of Uzbekistan No. 330 dated November 12, 2015.