

**IDENTIFICATION OF THE COMPOSITION OF AN UNKNOWN
POWDER SUBMITTED FOR EXPERTISE USING (IQ)-
SPECTROPHOTOMETRY AND GAS CHROMATOGRAPHY-MASS
SPECTROMETRY METHODS**

Boisxo'jayeva Adibaxon Anvarovna,

Senior Expert at the Materials, Articles, and Objects Forensic Examination

Laboratory of the Republic Forensic Expertise Center named after X.

Sulaymonova

Xalilova Nilufar Shuxratillayevna,

Head of Department, Institute of Forensic Scientific Research, Republic Forensic

Expertise Center named after X. Sulaymonova, Candidate of Pharmaceutical

Sciences

Usmanalieva Zumrad Uktamovna,

Head of the Department of Analytical and Pharmaceutical Chemistry, Institute of

Pharmaceutical Education and Research, Doctor of Pharmaceutical Sciences,

Professor

Annotation:

The article discusses the material evidence presented for examination, the features of the study of unknown powders available in forensic chemical laboratories.

Key words: examination, Pregabalin, sample, method, gas chromatography-mass spectrometry, IQ-spectrophotometry, spectrum.

The prevalence of psychoactive and highly potent substances among young people in our country is increasing, along with traditional narcotics. In particular, the illegal consumption of psychoactive and highly potent substances by minors or unhealthy individuals is leading to the destruction of young families. Many different crimes are being committed by most young people. Although all the necessary

conditions are created in our society for our youth to grow up mentally and physically healthy, to acquire modern knowledge and professions, it is a sad fact that they are committing crimes related to such substances and are falling into the path of drug addiction. In order to prevent such evils and combat crime in our society, a lot of research is being conducted in the field of forensic expertise. One of these is the Pregabalin drug, which has been encountered many times in forensic practice in recent years. The study of micro quantities of evidence submitted for expertise in the range of 0.1-0.5 mg has a number of peculiarities. These features are related to the need to improve methods due to the very small amount of the substance, their variety of forms, the diversity of objects carrying the substances, the interaction of micro quantities of substances and carrier objects, and so on. Micro quantities of evidence are often used in the investigative process, in forensic practice, as a source of information about the details of a criminal case, because the nature of the substance in the submitted small amount of evidence or in the objects carrying it, the place where the micro-object was found, the type of carrier object provides important information for solving many questions. It has been experimentally established that the amount of evidence necessary for research as a solid substance is -1 mg, and as a liquid - 1 ml. The study of small amounts of evidence requires significant attention and effort from specialists, and the instruments and methods used to solve diagnostic and identification problems are subject to particularly stringent requirements. This is explained by the small amounts of these substances, their state and the unusual nature of their distribution in carrier objects, which makes their identification particularly complex. Determining the nature, group affiliation and origin of small amounts of evidence provides the investigator with important information about the circumstances of the case. [3].

Research Objective: "The objective of the research is to explore the feasibility of using highly sensitive and modern methods available at the Central Laboratory to determine the nature and composition of unknown substances or traces of substances found in carrier objects submitted for expert examination."

Forensic laboratories frequently receive requests from law enforcement agencies to analyze narcotic drugs, psychoactive substances, and potent drugs, and to determine their type and name. In one particular case, the Central Laboratory received a sealed package containing a small amount of white powdery substance found at the residence of citizen R. Soliev. The task given to the experts was to determine whether the evidence found at R. Soliev's residence - a small amount of white powdery substance in a plastic bag - contained narcotic drugs, psychoactive and potent substances, and if so, which category it belonged to.

The initial step in analyzing the sample submitted for expertise involved taking a 0.001 g sample of the white powdery substance submitted for testing, adding 2.0 ml of 96% ethyl alcohol solvent, and extracting at room temperature for 6 hours. The sample was then filtered and used for analysis.

The second stage of the study utilized two methods:

1. Infrared Spectroscopy (IR): While chromatographic analysis is the most common method for analyzing narcotic drugs, psychoactive and potent substances, in recent years, the IR spectroscopy method has also become effective due to its high information content, sensitivity, and selectivity in forensic chemical expertise, and is available in every forensic laboratory. IR spectroscopy has become a major method for studying substances of various chemical natures, including medicinal compounds and narcotic substances. The method is used in forensic analysis to determine the nature of objects of unknown origin and to prove the similarity or difference of substances with similar chemical structures (from the same series).

To determine the presence or absence of narcotic drugs, psychoactive and potent substances in the sample submitted for testing, the prepared dry residue was analyzed using an Agilent Technology FTIR-640 IR spectrophotometer under the following conditions: recording range 4000-400 cm^{-1} , scanning rate - 40..

2. Gas Chromatography-Mass Spectrometry (GC-MS): This method is one of the most widely used methods in the analysis of organic substances among

physicochemical methods, distinguished by its high sensitivity, accuracy, and the ability to identify very small quantities of the substance under study in complex mixtures. The method is also widely used to identify substances of unknown origin, in the absence of their standard samples, and to identify metabolites formed from toxic substances as a result of their metabolism in the body. [1]

The analysis was performed on a gas chromatograph-mass spectrometer (GC-MS) from "Agilent Technology" GS 6890/MS 5973N (capillary column, 30 m long, 0.25 mm diameter, 5% phenylmethylsiloxane, mass selective detector) under the following analytical conditions:

- Electron ionization energy: 70 eV
- Injector temperature: 280°C
- Oven temperature: 150°C to 180°C, programmed temperature rise rate of 15°C per minute
- Sample volume: 1 μ L
- Analyte vapor pressure: 10 mmHg
- Analysis duration: 27 minutes
- Carrier gas: Hydrogen, flow rate: 2.4 mL/min.

RESULTS AND DISCUSSIONS

1. Under the conditions described above for IR spectrophotometry, the IR spectrum of the unknown substance revealed characteristic absorption bands at 2954, 2600, 1643, 1545, 1387, 1333, 700, 597, and 422 cm^{-1} , indicating vibrational modes. The number, intensity, and overall appearance of these absorption bands in the obtained spectrum matched the structure of Pregabalin (Pregabaline) present in the computer's spectral library. The IR spectrum of Pregabalin is shown below.

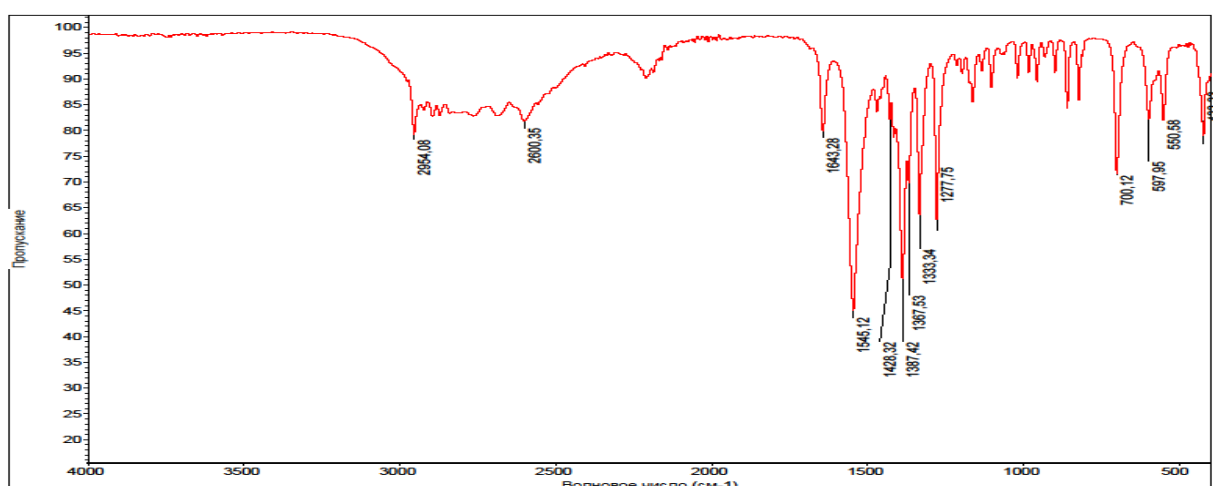


Figure 1. IR Spectrum of Pregabalin

The results of the conducted research revealed the presence of Pregabalin in the analyzed sample, identified by its characteristic absorption bands and functional groups.

2. GC-MS Analysis:

To conduct the GC-MS analysis, 1 μ L of the prepared sample was injected into the chromatographic column. The results obtained are as follows::

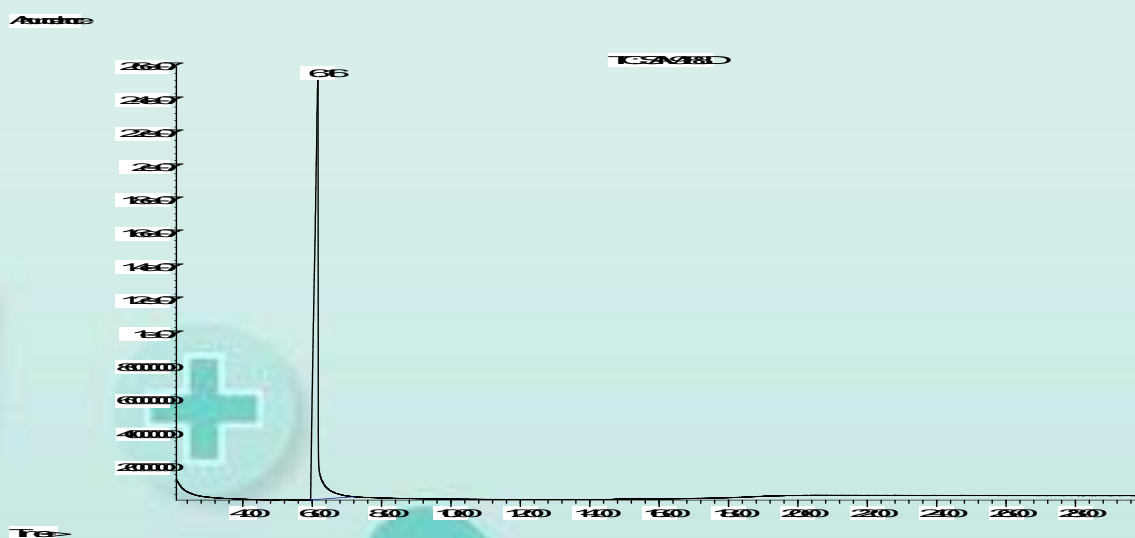


Figure 2. Chromatogram of Pregabalin

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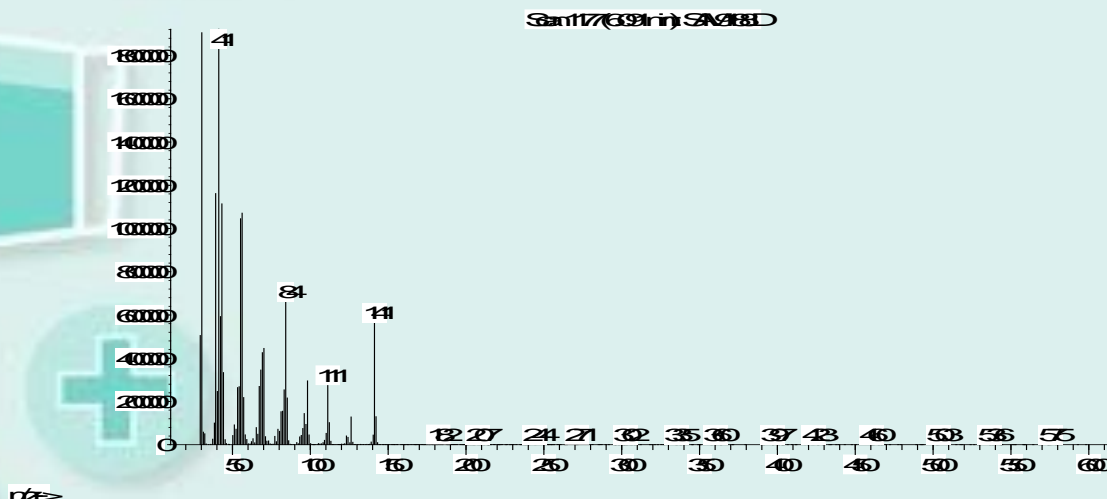
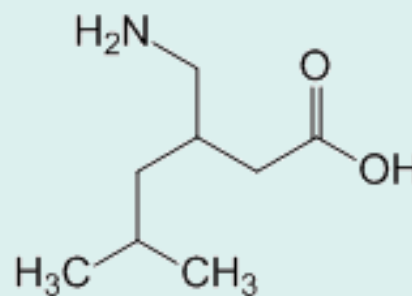


Figure 3. Mass Spectrum of Pregabalin

The peaks observed in the chromatogram and mass spectra of the sample were identified based on comparing their mass spectra with those in the NIST 02.L, NIST11.L, Wiley275.L, SWDRUG.L, CAYMANSPECTRA.L, and SWDRUG 3.5.L mass spectral databases. Peaks with a retention time of 6.16 minutes were detected in the gas chromatograph-mass spectrum of the sample, with molecular ions of m/z 46, 84, 111, and 141, indicating that the substance is Pregabalin. The analysis of the obtained chromatogram shows that the mass spectra of the investigated substance are characterized by the presence of specific ions formed by the fragmentation of molecular ions.

Thus, the gas chromatography-mass spectrometry analysis of the sample obtained from a small amount of white powdery substance in a plastic bag submitted for testing revealed the retention time, molecular and fragment ions, their intensity, and the specific fragmentation pattern of each molecular ion. These features can be used to determine the nature, type, and group affiliation of the Pregabalin micro-quantities present in the sample, and to address other criminal issues. [5].

Pregabalin (Pregabalinum)

(S)-3-(Aminometil)-5- methylgexanic acid Brutto**(C₈H₁₇NO₂)**

Pregabalin is a derivative of gamma-aminobutyric acid (GABA). It is a white solid crystalline substance, highly soluble in water. Its molecular mass is 159.23..

It is marketed under the brand names "Lyrica," "Gabana," "Algerika," "Neogabin," and "Linbag" and is used as an anticonvulsant for epilepsy. Pregabalin also has analgesic properties, making it a prescribed medication for patients with neuralgia. However, taking Pregabalin without a doctor's prescription can lead to dependence and turn users into passive addicts. They feel compelled to consume these drugs regularly to maintain mental and physical resilience. Consequently, this drug addiction has several adverse effects, including:

- Cardiovascular System: High blood pressure, heart failure, hyperemia attacks, and tachycardia.
- Respiratory System: Pulmonary edema, dryness of nasal mucosa, and nosebleeds.
- Gastrointestinal System: Nausea, diarrhea, pancreatitis, constipation.
- Musculoskeletal System: Muscle pain, inflammation of muscle tissue.
- Vision: Dryness of the conjunctiva, stinging.
- Kidney: Enuresis.
- Nervous System: Sleep disorders, increased depression, hallucinations, development of schizophrenia, and fatigue.

The most severe consequence of Pregabalin overdose is coma and even death due to the cessation of heart and kidney function. [6] Pregabalin is listed in the list of potent substances in Uzbekistan (position 55), according to the Resolution of the

Cabinet of Ministers of the Republic of Uzbekistan No. 818 of September 27, 2019,
"On Regulating the Circulation of Potent Substances in the Republic of Uzbekistan.

CONCLUSION

The analysis revealed that the white powdery substance found in the plastic bag submitted for testing contained Pregabalin. This substance was identified using modern, highly sensitive analytical techniques including infrared spectrophotometry, known for its simplicity and ease of sample preparation, and gas chromatography-mass spectrometry (GC-MS), characterized by high sensitivity and accuracy.

REFERENCES

1. Islomov T.Kh., Abdullaeva M.U. Forensic examination of drugs using chromatographic methods. Methodological Manual. - T., 1996. 150 p.
2. Islomov T.Kh., Abdullaeva M.U. Forensic examination of drugs by chromatography-mass spectrometry. In: Collection of scientific works of the Main Bureau of Forensic Medical Expertise of the Ministry of Health of the Republic of Uzbekistan, T., 2000, pp. 106-109.
3. Khalilova N.Sh., Korablyova N.V., Vetrova V.A., Abdullaeva M.U., Jalilov F.S. A method for forensic chemical examination of trace amounts of narcotic drugs using HPLC with a mass spectrometer detector. In: Proceedings of the International Scientific and Practical Conference, Ukraine, Kharkiv, 2019, pp. 205-216.
4. Abdullaeva M.U., Usmanalieva Z.U., Khalilova N.Sh., Boixojaeva A.A. "Expert method for examining trace amounts of the psychotropic substance pregabalin using gas chromatography with a mass spectrometer detector", In: Proceedings of the IV International Scientific and Practical Conference, Ukraine, Kharkiv, 2020, pp. 93-94.

5. Khalilova N.Sh. Methods for detecting trace amounts of substances or residues on various types of objects submitted for examination. Methodical recommendations for experts. - Tashkent, 2022. 32 p.
6. Khalilova N.Sh., Boisxo'jaeva A.A. Development of a scientifically based methodological guide for detecting residues of pregabalin and tramadol on various objects and its application in forensic practice. Methodical recommendations for experts. - Tashkent, 2020. 34 p.
7. Rasulova M. B., Muratov F. X., Rasulova D. K. Neuroimaging Indicators of Speech Disorders in the Prognosis of Aphasia Rehabilitation //British Medical Journal. – 2022. – T. 2. – №. 6.
8. Rasulova D., Rasulova M., Kuranbaeva S. The influence of various factors on the recovery dynamics of post-stroke aphasia //Journal of the Neurological Sciences. – 2019. – T. 405. – C. 152.