

ONE STEP PREPARATION OF BIOSAMPLES FOR GC-MS TOXICOLOGICAL ANALYSIS BASED ON THE EXTRACTIVE FREEZING-OUT

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The complex chemical composition and diversity of biological objects cause multistage toxicological analysis. At present for the determination of xenobiotics widely used GC-MS due to the high specificity and sensitivity. Preparation of samples is usually based on liquid and solid-phase extraction.¹

Objects of toxicological analysis, including biological liquids contain a lot of water, so it is convenient to use the method of extractive freezing-out (EF)²⁻³ as a stage of sample preparation. The combination of the EF with centrifugation (EFC) due to a significant reduction of the extractant share allows, as a result of one step procedure, 25-30-fold enrichment of analyte in the resulting extract.⁴⁻⁵ The EFC method, while retaining the advantages of Ef over solid-phase and liquid extraction, is even more effective in the analysis of dispersed samples. The EFC extracts contain almost no water. Therefore, bypassing dehydration, they can be immediately analyzed by gas chromatography without risk for the separation column. The use of the EFC method at the sample preparation stage of bioassays also allows to significantly reduce the background of endogenous coextractive substances due to the fact that peptides and carbohydrates remain in the aqueous solid phase of the sample.

The example of determination of caffeine, pyrovalerone⁶ and ketamine in biological liquids and tissues (liver, kidney from the corpse) found that extracts obtained by the EFC method, can be directly investigated using GC-MS. Thus, sample preparation consists of only one stage and does not exceed 25 min

References

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