A therapy monitored by drug concentration is a course of action aiming at dosing the patient with such amounts of the drug that the resulting concentrations fall within the therapeutic range, *i.e.* the concentration range at which a given medicine offers a high degree of therapeutic effectiveness and low risk of toxicities. Therefore the paramount aim of this project is a new approach to testing (in vitro and in vivo) of the metabolic biotransformation products of drugs from different therapeutic groups (antibiotics, cardiac medicines, immunosuppressants, antipsychotics) with the application of combined separation techniques. The studies carried out as a part of the project will focus on developing and optimizing the methods of isolation and determination of drugs and their metabolites based on the modern, efficient techniques of extraction, separation and spectrometry. The analysis of bioactive compounds that are the research subject will involve determination and identification of the selected drugs and their metabolites in authentic samples (blood serum, urine, blood and tissue). The research will have the following stages: sample preparation (based on implementing microsomal fraction enzymes of mouse, rat and human cells; electrochemical generation of potential metabolites with the use of spectroelectrochemical cell and clinical samples from patients); separation (with liquid chromatography) and identification (detection through mass spectrometry). The adopted methodology of metabolomic studies will apply to all the stages of the analytical procedure including bioinformatic data processing. This goal will be reached in several ways, namely through electrochemical stimulation of metabolism (biotransformation reactions of phase I and II) of selected drugs with the help of electrochemical reactor connected on line/off line to a mass spectrometer (QqQ MS, MALDI). Besides, the selected analytes will be transformed in vitro in the presence of metabolizing enzymes from the microsomal fraction of liver cells. What is more, the obtained results will be correlated with the data collected for authentic samples from research hospital patients. This topic was chosen because the current knowledge concerning metabolic pathways and metabolism of most frequently used bioactive compounds (antibiotics, antipsychotics, cardiovascular drugs and immunosuppressants) is crucial for explaining and determining the toxicity of a given substance and its mechanism of action in a human organism. The knowledge regarding drug metabolism is predominantly based on the results of in vivo tests performed on blood or urine samples obtained from patients. Development of selective and precise methods of determining and separating the chosen drugs belonging to different therapeutic groups will find application in metabolomics for the purposes of biomedical diagnostics. Such comprehensive approach has not yet been adopted towards the group of substances being the subject of this research. The results obtained will be indispensable for optimizing determination methods and for implementation of the developed methodologies in routine analysis of the selected drugs and their metabolites belonging to different therapeutic groups. These issues undoubtedly exceed the scope of analytical chemistry and require interdisciplinary approach to the posed research problem. Application of the developed analytical methodologies in medical diagnostics can be a driving force in more effective fight against the threats common nowadays, such as auto immunological and cardiovascular diseases, mental illnesses and bacterial infections as well as in diagnostics of festering wounds.