TESTING OF PALLADIUM BEFORE X-RAY FLUORESCENT DETERMINATION USING SORPTION PRECONCENTRATION

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The determination of palladium in ores and industrial products is necessary when searching deposits and monitoring of many technological processes. Usually highly sensitive instrumental methods are used for this purpose. However their usage for the analysis of large number of samples is often unfairly expensive. The cost of analysis may be lowered by application of simple express methods, for example of tests-methods and methods of molecular spectroscopy. Thus, it is possible to successfully combine sample preparation and determination stages obtaining analytical forms - colored compounds of palladium - directly on a phase of sorbent-concentrate.

The spectroscopic method for determination of palladium in solutions of complicated composition including sorption of metal on cellulose filters impregnated with tri-n-octylamine with the subsequent formation of colored compound of palladium with 4-(2-pyridylazo)resorcinol (PAR) on a filter is designed. The conditions for preconcentration of palladium are the same with that for the formation of colored compounds of metal. Due to this fact the complexes of palladium with PAR can be obtained directly in a phase of sorbent. Both complexes of palladium with PAR - green and red forms prepared accordingly in a strongly acidic and in weakly acidic - neutral media, - were used as analytical forms. The best selectivity was obtained with green form. In this case large amounts of accompanying elements (macrocomponents of ores) do not interfere the determination of palladium.

The kinetics of formation of palladium complexes with PAR in solution and in solid was studied. The following conditions are necessary to achieve the equilibrium: concentration of chloride and sulfuric acid 0.1 M and reaction time of 40 min.

Palladium was determined on the filters by diffuse reflectance spectroscopy both with the calculation of colorimetric characteristics and with the test-scale. The detection limits of palladium using spectrophotometer “SPECTROTON” are 0,5 (green form) and 0,3 (red form) μg of the element on a filter. High precision of results obtaining (RSD ≤ 0,05) is observed in a wide range of palladium concentration.

MATHEMATICAL MODELLING OF SOME ORGANIC MICROPOLLUTANTS RECOVERY ON THE HYDROPHOBIC SORBENTS


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The problem of natural waters pollution by various toxicants, like phenols or rocket fuel components, forces to use of high precision analytical methods including preconcentration. Solid phase extraction (SPE), especially performed in column mode, is the most promising technique for the preconcentration of organic substances from solutions, because it overcomes many disadvantages of liquid-liquid extraction.

For the choice of sorbents for SPE it is necessary to define strict criteria of sorption efficiency. The most integral criterion proposed in this work is the concentration efficiency (CE) determined as the sample flow rate on the preconcentration step under quantitative recovery of analyte. It was demonstrated that the maximum achievable CE has a single value for the given preconcentration factor and the recovery; therefore this criterion can be used for the comparison of different sorbents.

Mathematical modeling is most promising approach to description of SPE process and prediction of sorption efficiency. In present work using of phenomenological liquid film diffusion and solid diffusion models the relation between model parameters describing thermodynamic and kinetic properties of adsorption (distribution ratio, solid diffusion coefficient of sorbate in a sorbent phase, liquid film coefficient, dimension of the sorbent particles) and CE, as well as optimum SPE conditions (sorbent bed size and sample flow rate) has been established.

The adsorption of phenols on hexadecylsilica, styrene-divinylbenzene copolymers with various cross-linking degree and hyper cross-linked polystyrenes was investigated. The adsorption isotherms and dynamic breakthrough curves of phenol and catechol were obtained. The type of mass-transfer was determined and parameters of the model (distribution ratio, solid diffusion coefficient, liquid film coefficient) were calculated. To compare the sorbents investigated for the preconcentration of phenol and catechol, the values of maximum achievable CE were calculated. The group of hyper cross-linked polymeric sorbents possessing both high thermodynamic and kinetic parameters was found the most promising for the preconcentration of phenols.

Optimal conditions for SPE of 1,1-dimethylhydrazine derivate – 1,1-dimethylhydrazon of 4-nitrobenzaldehyde (DHB) on Cı6-silica were chosen. In this case choice of sorbent is not critical, because of high distribution ratio due to hydrophobic nature of this compound. The adsorption isotherms and dynamic breakthrough curves of DHB on Cı6-silica were obtained. To calculate optimum preconcentration conditions parameters of the model were determined. Bed sizes of 20X4 mm i.d. and flow rate 4.2 ml/min were chosen. Under these conditions the quantitative recovery of analyte from 120 ml of sample and enrichment factor of 400 were achieved.

THIN-LAYER FILTERS FOR PRECONCENTRATION OF GOLD AND PALLADIUM FROM SOLUTIONS

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Key words: sorption preconcentration, filters, noble metals, analysis of ores and alloys

One of the promising ways for determination of small amounts of noble metals in solutions of complicated composition is working out of the combined methods of analysis including preconcentration stage. Dynamic sorption preconcentration that provides high enrichment factors and does not require separation of phases is mostly effective.